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for
TESTING MATERIALS

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PROCEEDINGS
of the
FORTY-SIXTH ANNUAL MEETING

Held at Pittsburgh, Pa.

June 28—July 1, 1943

VOLUME 43

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TECHNICAL PAPERS

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SUMMARY OF PROCEEDINGS OF THE FORTY-SIXTH ANNUAL MEETING

PITTSBURGH, PA., JUNE 28–JULY 1, 1943

The following Summary records the action taken by the Society at the Annual Meeting on the Recommendations Affecting Standards and Tentative Standards appearing in the annual reports of the standing committees. In the individual committee reports attention is called through an editorial footnote to any modifications of the recommendations or where the recommendations of the committee were not approved. In all other instances it is to be understood that the recommendations were approved as submitted by the committee. The new and revised standards and tentative standards are published in the 1943 Supplement to Book of A.S.T.M. Standards, Parts I, II, and III. Except as noted, the reports and paper appear in this volume.

This summary of the Forty-sixth Annual Meeting of the American Society for Testing Materials, held at Hotel William Penn, Pittsburgh, Pa., June 28–July 1, 1943, is a record of the transactions of the meeting including the actions taken on the various recommendations submitted by the standing committees. In all, 15 sessions were held.

The registered attendance of the meeting is as follows: Members present or represented, 757; committee members, 323; guests, 372; total, 1452.

The Proceedings are set forth session by session. There were 51 reports and 52 formal papers presented. The record with respect to each has been briefed, the recommendations in the reports having been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentative standards and proposed revisions of existing standards, and as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentative standards as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported as "adopted as standard," or "adopted as standard, revisions in" it is understood that this indicates approval of the annual meeting for reference to letter ballot of the Society. The various recommendations so recorded are included in the Society letter ballot.[1] The actions designated as "accepted as tentative" or "accepted as tentative, revisions in," are self evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative.

While all of the items on the program are recorded under the particular session in which they were presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all the reports together with the page reference where the action thereon is recorded.

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[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, was canvassed on September 10, 1943, and all actions were approved.

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FIRST AND SECOND SESSIONS—SYMPOSIUM ON SOIL TEST METHODS

TUESDAY, JUNE 29, 9.30 A.M. AND 2 P.M.

SESSION CHAIRMAN: PAUL RAPP

President H. J. Ball, in opening the Forty-sixth Annual Meeting, extended a cordial welcome to all members and guests of the Society in attendance. He commented briefly on the growth of the Society and the considerable expansion in its activities since its organization forty-five years ago. In calling attention to the program for the technical sessions at which several symposiums and a large number of technical papers and annual reports of the committees would be presented, he stated that they represent many contributions to the war effort. He also referred to the many scheduled meetings of standing committees and their subcommittees at which other work was being furthered and plans made for new programs of activity many of which would be of direct assistance to the war effort.

Committee D-18 on Soils for Engineering Purposes:

Report presented by W. R. Nelson, Secretary.

Symposium on Soil Test Methods:

This symposium is essentially a preliminary symposium or discussion looking toward the holding of a formal symposium on testing of soils, under the sponsorship of Committee D-18 on Soils for Engineering Purposes, at a later annual meeting.

In connection with the present symposium, a number of individuals had submitted descriptions of various soil test procedures with which they were familiar. These had been put in standard form and were presented on behalf of Committee D-18 by Paul Rapp of the Public Roads Administration acting for F. C. Lang of the Minnesota Highway Dept., chairman of the symposium committee. In this presentation, he was assisted by the following individuals who presented in somewhat greater detail the specific tests grouped as follows: Indication Tests—C. A. Hogentogler, Public Roads Administration; Compaction Test—K. B. Woods, Purdue University; Shear Test—E. J. Kilcawley, Rensselaer Polytechnic Institute; Test for Bituminous Mixtures—F. V. Reagel, Missouri State Highway Department; and Test for Soil Cement Mixtures—M. D. Catton, Portland Cement Association.

It is proposed that the individual tests will be reviewed in the light of discussion submitted and issued in compilation form as information preparatory to holding the formal symposium on soil testing at some future annual meeting of the Society.

THIRD SESSION—WATER

TUESDAY, JUNE 29, 8 P.M.

SESSION CHAIRMAN: R. E. HALL

Committee D-19 on Water for Industrial Uses:

Report presented by Max Hecht, chairman, and the following actions taken:

Adopted as Standard:

Method for Determination of Total Carbon Dioxide and Calculation of the Carbonate and Bicarbonate Ions in Industrial Waters (D 513 - 38 T), as revised

Adopted as Standard (Continued):

Method for Determination of the Total Orthophosphate and Calculation of the Respective Orthophosphate Ions in Industrial Waters (D 515 - 40 T), as revised

Joint Research Committee on Boiler Feedwater Studies:

Report, not preprinted, presented from manuscript by C. H. Fellows, chairman.

Papers:

The following papers were presented:

Laboratory and Field Methods for the Determination of Dissolved Oxygen—Robert C. Adams, Robert E. Barnett, and Daniel E. Keller, Jr., presented by Mr. Adams.

Applicability of the Schwartz-Gurney Method for Determining Dissolved Oxygen in Boiler Feedwater and Modification of the Method to Make It Applicable Especially in the Presence of Such Impurities as Are Encountered in Power Plants—R. C. Ulmer, J. M. Reynar, and J. M. Decker, presented by Mr. Reynar.

Symposium on the Identification of Water-Formed Deposits, Scales, and Corrosion Products by Physico-chemical Methods:

The symposium was introduced by H. C. Miller, and the following papers were then presented:

X-ray Diffraction Methods in the Study of Power Plant Deposits—C. E. Imhoff and L. A. Burkhardt, presented by Mr. Imhoff.

Diagnosis of Water Problems at Limbo Station—Everett P. Partridge, R. K. Scott, and P. H. Morrison, presented by Mr. Partridge.

The Interpretation of Analyses and Problems Encountered in Water Deposits—J. A. Holmes and A. O. Walker, presented by Mr. Holmes.

FOURTH SESSION—HARDNESS SYMPOSIUM

TUESDAY, JUNE 29, 8 P.M.

SESSION CO-CHAIRMAN: J. M. LESSELS; R. E. PETERSON

Committee E-1 on Methods of Testing:

Report presented by W. H. Fulweiler, chairman.

In presenting the report, Mr. Fulweiler called upon H. F. Moore to describe the activities of Technical Committee I on Mechanical Testing, and on E. C. Bingham to report on the work of Technical Committee II on Consistency, Plasticity, and Related Properties.

Symposium on the Hardness Test in Relation to Design

The symposium was introduced by J. M. Lessells, after which the following papers covering features of the subject were presented:

Present Types of Hardness Tests—S. R. Williams, presented from manuscript by the author.

Fundamentals of Hardness Testing—S. L. Hoyt, presented from manuscript by the author.

The meeting was then thrown open to general discussion, in which a number of those present participated.

FIFTH SESSION—PETROLEUM PRODUCTS, ASPHALT

WEDNESDAY, JUNE 30, 9.30 A.M.

SESSION CO-CHAIRMEN: A. T. GOLDBECK; F. V. REAGEL

Committee D-2 on Petroleum Products and Lubricants:

Report presented by T. A. Boyd, chairman, and the following actions taken:

Accepted as Tentative:

Test for Oil Content of Paraffin Wax, with a correction in Section 1 changing the "10 per cent" limit of oil in wax to read "15 per cent."

Accepted as Tentative, Revisions in:

Test for Aniline Point of Petroleum Products (D 611 - 41 T), with the following additional revision, not preprinted:[2]

Section 3 (c).—Omit the words "variable speed" in the description of the mechanical stirrer.

Figure 1.—Omit the words "variable speed" before the word "motor" in the identification of parts in this figure.

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[2] This recommendation, not preprinted, was accepted by unanimous consent, subject to letter ballot of Committee D-2, which ballot has been favorable and the results are given in the report of Committee D-2, see p. 262.

Test for Ignition Quality of Diesel Fuels (D 613 - 41 T)
Test for Knock Characteristics of Aviation Fuels (D 614 - 42 T)
Test for Knock Characteristics of Motor Fuels (D 357 - 42 T)
Test for Saponification Number of Petroleum Products by Color-Indicator Titration (D 94 - 42 T), submitted jointly with Committee D-9 on Electrical Insulating Materials
Test for Ash Content of Petroleum Oils (D 482 - 38 T), with the following modification, not preprinted:[2]

Section 1.—Add the following note to this section:

NOTE.—When this method is applied to used oils from engines operating on leaded fuels and to new and used oils containing metallo-organic addition agents, large portions of the metal compounds may be lost by vaporization. In such cases the ash is not a measure either of inorganic contaminating material or of the metallo-organic compounds in the oil. This method is not recommended for use with such oils.

Adopted as Standard:

Test for Carbonizable Substances in Paraffin Wax (D 612 - 41 T), as revised
Test for Carbonizable Substances in White Mineral Oil (Liquid Petrolatum) (D 565 - 41 T)

Adopted as Standard, Revisions in:

Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 42)
Viscosity-Temperature Charts for Liquid Petroleum Products (D 341 - 39)

Accepted for Publication as Information Only:

Test for Saponification Number of Petroleum Products by Electrometric Titration
Test for Oxidation Characteristics of Steam-Turbine Oils

Sectional Committee Z11 on Petroleum Products and Lubricants:

Report, not preprinted, presented from manuscript by T. A. Boyd, chairman.

Committee D-8 on Bituminous Waterproofing and Roofing Materials:

Report presented by E. H. Berger, vice-chairman, and the following action taken:

Editorial Change Accepted in:

Spec. for Asphalt for Dampproofing and Waterproofing (D 449 - 42 T), the following editorial change, not preprinted, was accepted by unanimous consent:

Section 4.—Change "Type A" to read "Type B" and change "Type B" to read "Type A," making a corresponding change in Table I on physical properties of asphalt, in order to conform with the type designations in the Standard Specifications for Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (D 450 - 41).

Sectional Committee A37 on Road and Paving Materials:

Report presented, in the absence of the chairman, by the chair.

Committee D-4 on Road and Paving Materials:

Report presented by C. N. Forrest, chairman, and the following actions taken:

Adopted as Standard:

Spec. for Slow-Setting Emulsified Asphalts (for Fine Aggregate Mixes) (D 631 - 41 T)
Spec. for Asphalt Filler for Brick Pavements (D 241 - 39 T)
Spec. for Sodium Chloride (D 632 - 41 T)[3]

Papers:

The following papers were presented:

A Method for Evaluating Performance in Service of Slow-Curing Asphalts[4]—Joseph Zapata, presented by the author.
Accelerated Weathering of Bituminous Materials—Effect of Operating Variables—Bruce Weetman, presented by the author.
A Method for the Testing and Evaluation of Road Tars—E. O. Rhodes and H. E. Gillander, presented by Mr. Rhodes.

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[3] This recommendation, not preprinted, was accepted by unanimous consent, subject to letter ballot of Committee D-4, which ballot has been favorable and the results are given in the report of Committee D-4, see p. 285.

[4] Published in ASTM BULLETIN, No. 124, October, 1943, p. 31.

SIXTH SESSION—CORROSION, ELECTRODEPOSITED COATINGS

WEDNESDAY, JUNE 30, 9.30 A.M.

SESSION CO-CHAIRMEN: W. H. FINKELDEY; E. A. ANDERSON

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented by Sam Tour, chairman, and the following action taken:

Accepted as Tentative:

Method of Total Immersion Testing of Non-Ferrous Metals

Committee A-5 on Corrosion of Iron and Steel:

Report presented by C. D. Hocker, chairman, and the following action taken:

Adopted as Standard, Revision in:

Spec. for Zinc-Coated (Galvanized) Iron or Steel Telephone and Telegraph Line Wire (A 111 - 33):[5]

Section 16 (c).—Change the first sentence to read as follows by the addition of the italicized figures and the omission of those in brackets:

> Each coil shall be securely bound in four separate places, equally spaced, with at least two wrappings of galvanized wire, not smaller than [0.109] *0.080* in. in diameter.

Tentative Standards Continued as Tentative Without Revision:

Spec. for Zinc-Coated (Galvanized) Iron or Steel Sheets (A 93 - 38 T)
Spec. for Electrodeposited Coatings of Zinc on Steel (A 164 - 40 T)
Spec. for Electrodeposited Coatings of Cadmium on Steel (A 165 - 40 T)
Test for Local Thickness of Electrodeposited Coatings (A 219 - 40 T), presented jointly with Committee B-8 on Electrodeposited Metallic Coatings

Committee B-8 on Electrodeposited Metallic Coatings:

Report presented by E. A. Anderson, vice-chairman, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Preparation of Low-Carbon Steel for Electroplating

Tentative Standards Continued as Tentative Without Revision:

Spec. for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 - 41 T)
Spec. for Electrodeposited Coatings of Nickel and Chromium on Copper and Copper-Base Alloys (B 141 - 41 T)
Spec. for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys (B 142 - 41 T)
Test for Local Thickness of Electrodeposited Coatings (A 219 - 40 T), presented jointly with Committee A-5 on Corrosion of Iron and Steel

The paper by E. M. Baker, deceased, on "Factors Underlying Specifications for Electrodeposited Metallic Coatings," appended to the report, was presented by E. A. Anderson.

Papers:

The following papers were presented:

Atmospheric Corrosion of Copper—A. W. Tracy, D. H. Thompson and John R. Freeman, presented by Mr. Tracy.
The Total Immersion Corrosion Test—W. A. Wesley, presented by the author.
Chromium Plate in Engineering Applications—Its Thickness and Finishing—T. G. Coyle, presented by title.
Properties of Plated Lead Coatings on Steel—Gustaf Soderberg, presented by the author.
Use and Misuse of the Salt Spray Test as Applied to Electrodeposited Metallic Finishes[6]—C. H. Sample, presented by the author.

SEVENTH SESSION—TEXTILES, SOAPS, FIRE TESTS, GLASS, THERMAL INSULATING MATERIALS, FUELS

WEDNESDAY, JUNE 30, 1.30 P.M.

SESSION CO-CHAIRMEN: PRESIDENT H. J. BALL; W. A. SELVIG

Committee D-13 on Textile Materials:

Report presented by W. H. Whitcomb, secretary, and the following actions taken:

Accepted as Tentative:

Testing and Tolerances for Rope (Leaf and Bast Fibers)
Testing and Tolerances for Spun, Twisted, or Braided Products Made from Flax, Hemp, Ramie, or Mixtures Thereof
Test for Air Permeability of Textile Fabrics
Def. of Terms Relating to Textiles (D 123 - 43 T)
Test for Commercial Weight of Rayon Staple (D 540 - 43 T)

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[5] This recommendation, not preprinted, was accepted by unanimous consent, subject to letter ballot of Committee A-5, which ballot has been favorable and the results are given in the report of Committee A-5, see p. 71.

[6] Published in ASTM BULLETIN, No. 123, August, 1943, p. 19.

Accepted as Tentative, Revisions in:

Methods for Identification of Fibers in Textiles (D 276 - 41 T)
Methods of Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 - 42)
Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 - 42)
Methods of Testing and Tolerances for Spun Rayon Yarns and Threads (D 507 - 42)
Spec. and Methods of Test for Asbestos Tape for Electrical Purposes (D 315 - 41)

Adopted as Standard:

Test for Hard Scoured Wool in Wool in the Grease (Laboratory Scale Operations) (D 584 - 41 T)
Methods of Testing and Tolerances for Woven Glass Tubular Sleeving and Braids (D 581 - 40 T)
Methods of Testing and Tolerances for Woven Glass Fabrics (D 579 - 41 T), as revised
Methods of Testing and Tolerances for Woven Glass Tapes (D 580 - 41 T), as revised

Adopted as Standard, Revisions in:

Methods of Testing and Tolerances for Yarns Spun from Wool Mixed with Fibers Other than Wool (D 508 - 39)

It was announced that the recommendation in the preprinted report for the publication as tentative of the Test for Yarn Numbers in Woven Fabrics had been withdrawn by the committee.

Committee D-12 on Soaps and Other Detergents:

Report presented, in the absence of the chairman, by the chair and the following actions taken:[7]

Accepted as Tentative:

Test for Water-Immiscible Organic Solvents Volatile with Steam in Sulfonated and Sulfated Oils (D 500 - 43 T)

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Soaps and Other Detergents (D 459 - 42 T), by omission of the definition for straight soap, revisions in eight of the present definitions, and the addition of five new definitions.

Spec. for Trisodium Phosphate (D 538 - 39 T), with the following modifications, not preprinted:

Section 1.—Change to read as follows by the omission of the words in brackets:

> 1. These specifications [for trisodium phosphate] cover material suitable for various washing, cleaning, and scouring processes, with or without soap as conditions demand, and where a moderately strong alkaline material is desired.

Section 3.—In the table of requirements for chemical composition of trisodium phosphate, change the requirements for trisodium phosphate and phosphoric anhydride to read as follows by the addition of the italicized figures and the omission of those in brackets:

Trisodium phosphate calculated as $Na_3PO_4 \cdot 12H_2O$, from P_2O_5, min., per cent	[98]	*97*
Phosphoric anhydride (P_2O_5), min., per cent	[18.3]	*18.1*

Section 4 (a).—Change the percentage of trisodium phosphate from "98" to "97" where it occurs in the text and in the formula.

Adopted as Standard:

Methods of Sampling and Chemical Analysis of Special Detergents (D 501 - 41 T)

Editorial Changes Accepted in:

Spec. for Palm Oil Solid Soap (Type A, Pure; Type B, Blended) (D 535 - 41)
Spec. for Palm Oil Chip Soap (Type A, Pure; Type B, Blended) (D 536 - 42)
Spec. for Olive Oil Solid Soap (Type A, Pure; Type B, Blended) (D 592 - 42)
Spec. for Olive Oil Chip Soap (Type A, Pure; Type B, Blended) (D 630 - 42)

In the above four specifications, the changes, not preprinted, comprise the substitution of the word "straight" in place of "pure" in the titles and throughout the text of the specifications.

The paper on "Cooperative Studies on a Laboratory Method for Evaluating Synthetic Detergents," by J. B. Crowe, appended to the report, was presented by title.

Committee C-5 on Fire Tests of Materials and Construction:

Report presented by R. P. Miller, chairman, and the following actions taken:

Tentative Standard Continued as Tentative Without Revision:

Test for Fire-Retardant Properties of Wood (C 160 - 41 T)

Withdrawal of Tentative Standard:

Test for Fire-Retardant Properties of Wood for Scaffolding and Shoring (C 132 - 40 T)

The recommendation which appeared in the preprinted report for acceptance as tentative of the Methods of Fire Tests of Window Assemblies was discussed. This recommendation failed to receive the necessary two-thirds affirmative vote and on motion the proposed methods were referred back to Committee C-5 for further consideration.

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[7] These recommendations, not preprinted, were accepted by unanimous consent, subject to letter ballot of Committee D-12, which ballot has been favorable and the results are given in the report of Committee D-12, see p. 376.

Committee C-14 on Glass and Glass Products:

Report presented, in the absence of the chairman, by the chair and the following actions taken:

Adopted as Standard:

Chemical Analysis of Glass Sand (C 146 - 39 T)
Hydrostatic Pressure Test on Glass Containers (C 147 - 40 T), as revised
Polariscopic Examination of Glass Containers (C 148 - 40 T), as revised
Thermal Shock Test on Glass Containers (C 149 - 40 T), as revised
Flexure Testing of Glass (Determination of Modulus of Rupture) (C 158 - 40 T), as revised
Chemical Analysis of Soda-Lime Glass (C 169 - 41 T), as revised

Committee C-16 on Thermal Insulating Materials:

Report presented by E. T. Cope, chairman.

Committee D-3 on Gaseous Fuels:

Report presented by A. W. Gauger, chairman.

Committee D-5 on Coal and Coke:

Report presented by A. C. Fieldner, chairman, and the following actions taken:

Accepted as Tentative:

Test for Free-Swelling Index of Coal

Accepted as Tentative, Revisions in:

Method of Sampling Coals Classed According to Ash Content (D 492 - 40 T)
Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 42):[8]

Section 11.—Add the following note to this section:

NOTE 3.—Difficulty may be experienced in securing satisfactory check determinations of ash in the same or different laboratories for coals unusually high in calcite and pyrite. This is caused by varying amounts of sulfate sulfur being retained in the ash. When such difficulty is encountered, or when coals of relatively high ash content whose mineral matter composition is unknown are encountered, the ash should be determined by the following modified procedures:

(*a*) Place the porcelain capsules containing the dried coal from the moisture determination in a cold muffle furnace and heat gradually so that the temperature reaches 500 C. in 1 hr., and 750 C. in 2 hr. Heat to constant weight at 750 C. By this means pyritic sulfur will be oxidized and expelled before the calcite is decomposed. An ample supply of air in the muffle must be assured at all times to insure complete oxidation of the pyritic sulfur and proper circulation through the muffle must be assured to remove the SO_2 formed.

(*b*) The modified procedure described in Paragraph (*a*) should be adequate for determining ash in all troublesome commercial samples. However, samples may be encountered in certain special studies whose ash values are quite high and whose mineral matter contains much greater than normal amounts of calcite and pyrite. In such cases sulfate sulfur should be determined on the ash obtained by the modified cold muffle method and the value properly corrected, or the Parr[a] sulfated ash method as modified by Rees[b] should be used.

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[a] S. W. Parr, "Chemical Study of Illinois Coal," Bulletin No. 3, p. 35, Illinois Coal Mining Investigations, State Geological Survey, Urbana, Ill. (1916).

[b] O. W. Rees, "Determining Ash in High Carbonate Coals. Study of the Modified Method," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 9, pp. 307-309 (1937).

Adopted as Standard, Revisions in:

Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 42)

Accepted for Publication as Information Only:

Test for Expansion Pressure of Coal During Coking
Test for Plastic Properties of Coal by the Davis Type Plastometer
Test for Plastic Properties of Coal by the Gieseler Type Plastometer
Test for Carbonization Pressure of Bituminous Coal
Method for Measurement of Pressures Developed During Carbonization of Coal by the Movable Wall Oven
Test for Expansion Properties of Coal for Use in By-Product Coke Ovens by Means of the Bethlehem Test Oven
Test for Pressures, Strains, and Other Properties Developed During Carbonization of Coal[9]

Papers:

The following papers were presented:

Tests of the Fire Resistance and Thermal Properties of Solid Concrete Slabs and Their Significance—Carl A. Menzel, presented from manuscript by the author.
The Gieseler Method for Measurement of the Plastic Characteristics of Coking Coals—Glenn C. Soth and Charles C. Russell, presented by Mr. Soth.

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[8] This recommendation, not preprinted, was accepted by unanimous consent, subject to letter ballot of Committee D-5, which ballot has been favorable and the results are given in the report of Committee D-5, see p. 291.

[9] This proposed method was presented in connection with the discussion of the report by V. J. Altieri, see p. 320.

EIGHTH SESSION—NON-FERROUS METALS

WEDNESDAY, JUNE 30, 1.30 P.M.

SESSION CHAIRMAN: DEAN HARVEY

Committee B-1 on Copper and Copper-Alloy Wires for Electrical Conductors:

Report presented, in the absence of the chairman, by Alfred Bellis.

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented, in the absence of the chairman, by the chair, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Brass Sheet and Strip (B 36 - 42 T)
Spec. for Seamless Copper Tubes (B 75 - 41 T)
Spec. for Leaded Brass Sheet and Strip (B 121 - 42 T)
Spec. for Cartridge Brass Cartridge Cast Cups (B 129 - 42 T)
Spec. for Gilding Metal Sheet and Strip (B 130 - 42 T)
Spec. for Gilding Metal Bullet Jacket Cups (B 131 - 42 T)
Spec. for Miscellaneous Brass Tubes (B 135 - 42 T)
Spec. for Phosphor Bronze Wire (B 159 - 42 T)

Adopted as Standard:

Spec. for Copper Pipe, Standard Sizes (B 42 - 41 T), as revised
Spec. for Seamless Copper Tubing, Bright Annealed (B 68 - 42 T), as revised
Spec. for Manganese Bronze Rods, Bars, and Shapes (B 138 - 41 T)
Spec. for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 - 41 T)

Adopted as Standard, Revisions in:

Spec. for Rolled Copper-Alloy Bearing and Expansion Plates for Bridges and Other Structural Uses (B 100 - 40)
Spec. for Phosphor Bronze Sheet and Strip (B 103 - 42)
Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 42)

Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys:

Report presented by F. E. Bash, secretary, and the following actions taken:

Accepted as Tentative:

Test for Density of Fine Wire and Ribbon for Electronic Devices
Test for Effect of Controlled Atmospheres Upon Alloys in Electric Furnaces
Method for Life Test of Electrical Contact Materials, with the following revisions:

Sections 4 and 5.—Change from the form as preprinted to read as follows:

4. *Adjustment of Testing Machine.*—The machine shall be adjusted to give the following values:

Closing force, F1, for fine silver	100 g.
Closing force, F1, for other materials	[a]
Opening force, F2, for fine silver	75 g.
Opening force, F2, for other materials	[a]
Contact speed, opening and closing	1½ in. per sec.
Complete operation of each contact pair	60 per min.
Load current at 115 ± 5 v., 60 cycle a.c., unity power factor for fine silver	22 amp.
Load current at 115 ± 5 v., 60 cycle a.c., unity power factor for other materials	[a]

The periodic measurements (see Section 6 (*i*)) shall be made at intervals of 50,000 and 100,000 operations of each contact.

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[a] Under consideration by the committee.

5. *Preparation of Test Specimens.*—(*a*) Three sets of contacts shall be prepared for test specimens The contacts shall be soldered to nickel plated machine screws of S.A.E. No 1032 steel, approximately ½ in. in length. The contact material and head of the screw shall be machined to the desired diameter The contact material shall be ¼ in. in diameter and 3/32 in. in thickness. The threaded end of the screw shall be rounded to a 1/16-in. radius.

(*b*) The surface of the moving contact shall be crowned to a radius two and a half times the diameter of the contact. The stationary contact shall have a flat surface. Both contacts shall have a high initial polish.

(*c*) A hole 0.024 in. in diameter, 1/32 in. in depth, shall be drilled at the base of the contact material where it is joined to the steel screw.

Editorial Change Accepted in:

Method of Test for Temper of Strip and Sheet Metals for Electronic Devices (Spring-Back Method) (B 155 - 41 T)

Accepted for Publication as Information Only:

Method of Measurement of Equivalent Yield Stress of Thermostat Metals

It was announced that the recommendation in the preprinted report for the publication as tentative of the Specifications for Chromium-Nickel-Iron Alloy Castings for High-Temperature Service in Electric Furnaces had been withdrawn by the committee.

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by J. R. Townsend, chairman, and the following actions taken:

Adopted as Standard:

Spec. for Zinc-Base Alloy Die Castings (B 86 - 41 T)

Tentative Standards Continued as Tentative Without Revision:

Spec. for Magnesium-Base Alloy Die Castings (B 94 - 40 T)
Spec. for Lead- and Tin-Base Alloy Die Castings (B 102 - 39 T)

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by Sam Tour, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Aluminum Sheet and Plate for Use in Welded Pressure Vessels, with the following change:

Section 3.—Add the following to the table of requirements as to chemical composition:

Total of all elements except aluminum, max., per cent.................................... 1.0

Accepted as Tentative, Revisions in:

Spec. for Aluminum Sheet and Plate (B 25 - 42 T), with the following additional change:

Section 3.—Make the same change in this section as recommended above in Section 3 of the new Tentative Specifications for Aluminum Sheet and Plate for Use in Welded Pressure Vessels.

Spec. for Aluminum-Base Alloy Castings (B 26 - 42 T)
Spec. for Aluminum-Manganese Alloy Sheet and Plate (B 79 - 42 T)
Spec. for Aluminum-Base Alloy Permanent Mold Castings (B 108 - 41 T)
Spec. for Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 - 42 T)
Spec. for Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 - 42 T)

The paper by J. D. Edwards on "A Comparison of the Performance of Anodic Coatings on Wrought Aluminum Alloys When Exposed to Salt Spray and to the Weather," appended to the report, was presented by the author.

Committee B-2 on Non-Ferrous Metals and Alloys:

Report presented by E. E. Thum, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Oxygen-Free Electrolytic Copper Wire Bars, Billets, and Cakes (B 170 - 42 T), with the exception that the following proposed footnote, which appeared in the report as preprinted, was withdrawn by the committee:

> 0.15328 internal ohm (meter, gram) is regarded as an expression synonymous with exactly 100 per cent conductivity, and is a matter of definition, and the five places of decimals quoted above, or given as other permissible values of resistivity, do not imply that limits of accuracy to the fifth significant figures must be achieved in the determination of acceptance tests.

Adopted as Standard:

Spec. for Electrolytic Cathode Copper (B 115 - 41 T) without revision, the proposed footnote which appeared in the report as preprinted, as given above with respect to Specifications B 170 - 42 T, having been withdrawn by the committee.
Spec. for Pig Lead (B 29 - 40 T)
Spec. for Phosphor Copper (B 52 - 38 T)
Spec. for Silicon Copper (B 53 - 38 T)
Spec. for Brazing Solder (B 64 - 38 T)

Adopted as Standard, Revisions in:

Spec. for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 - 42)

Committee E-3 on Chemical Analysis of Metals:

Report presented by G. E. F. Lundell, chairman, and the following action taken:

Accepted as Tentative:

Rec. Practice for Apparatus and Reagents for Chemical Analysis of Metals

Committee E-2 on Spectrographic Analysis:

Report, not preprinted, presented from manuscript by H. V. Churchill, chairman.

Papers:

The following papers were presented:

Lead-Alloy Coated Copper Wire for Electrical Conductors—C. J. Snyder, presented by the author.
Experiments on Plastic Bending for Aluminum Alloy 17-T—Joseph Marin and F. D. Cotterman, presented by Dean Harvey.

Conservation of Tin in Soft Solders[10]—D. L. Colwell and W. C. Lang, presented by Mr. Lang.

The Testing and Properties of Low-Tin Solders Listed in Recent Federal Specifications—J. A. Kies and W. F. Roeser, presented by Mr. Kies.

NINTH SESSION—MARBURG LECTURE

WEDNESDAY, JUNE 30, 4.30 P.M.

SESSION CHAIRMAN: PRESIDENT H. J. BALL

In introducing the Eighteenth Edgar Marburg Lecturer, President Ball stated that the lecture has been designated with the name of Edgar Marburg to honor and perpetuate the memory of the first Secretary-Treasurer of the Society, and that it also serves to remind the present membership of the firm foundation he established which has brought such wide recognition to the A.S.T.M. as an outstanding forum for the discussion of properties and tests of engineering materials.

He then presented L. J. Markwardt, Chief, Division of Timber Mechanics, U. S. Forest Products Laboratory, who had chosen as the subject of his lecture "Wood as an Engineering Material." Mr. Markwardt presented a comprehensive picture of the many uses of wood in engineering construction and discussed in detail the marked advancement in its many applications that has taken place during recent years.

President Ball, in expressing appreciation to Mr. Markwardt for his fine exposition on a subject of particular interest to the A.S.T.M., presented to him on behalf of the Society, the Edgar Marburg Lecture Certificate.

TENTH SESSION—ADDRESSES: PRESIDENT BALL, FRANK B. BELL, AND ANDREW H. PHELPS; AWARD OF DUDLEY MEDAL

WEDNESDAY, JUNE 30, 8 P.M.

SESSION CHAIRMAN: PAST-PRESIDENT H. H. MORGAN

Committee E-10 on Standards:

This report, presented by J. R. Townsend, chairman, contained a complete list of the recommendations accepted by Committee E-10 on behalf of the Society during the year and also discussed considerations being given to organizing new standing committees on industrial aromatic hydrocarbons, metal powders and metal powder products, and adhesives.

Committee E-9 on Research:

In presenting this report, not preprinted, G. F. Jenks, chairman, discussed briefly the importance of research during present war time conditions and the part it will play in the postwar period. He called particular attention to the work on methods of testing being done by Committee D-20 on Plastics and to the activities of the newly formed Advisory Committee on Corrosion as well as to the cooperative research activities being carried on with the War Metallurgy Committee and other branches of Government research.

Executive Committee:

This report, presented by Secretary-Treasurer C. L. Warwick, discussed the basic wartime policy of A.S.T.M. and the Society's many contributions to the war effort. Information was also presented with respect to general Society activities, membership, publications, finances, administrative matters relating to committee activities, and cooperative activities with other engineering societies.

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[10] Published in ASTM BULLETIN, No. 123, August, 1943, p. 37.

Presidential Address:

The chairman then presented the President, H. J. Ball, who delivered the annual presidential address entitled "The Relation of the A.S.T.M. to the Textile Industry."[11]

Welcome by Pittsburgh District Committee:

The chairman then introduced A. R. Ellis, chairman of the Pittsburgh District Committee. On behalf of the Pittsburgh Committee, Mr. Ellis extended a cordial welcome to the Society membership and expressed the wish that the plans made by the local committee would contribute to a most successful Annual Meeting.

Address by Frank B. Bell:

Mr. Ellis then introduced Frank B. Bell, District Chief, Pittsburgh Ordnance District, who presented a talk on "Army Ordnance."[12]

Address by Andrew H. Phelps:

Mr. Ellis then presented Andrew H. Phelps, Vice-President in Charge of Purchases and Traffic, Westinghouse Electric and Manufacturing Co., who presented an address entitled "Maintaining Quality Under Wartime Conditions."[12]

Award of Dudley Medal:

The chairman then introduced Sabin Crocker, chairman of the Dudley Medal Committee, who presented, as recipient of the seventeenth award of the Charles B. Dudley Medal, Walter Bonsack, Chief Metallurgist, In Charge of Research Laboratories, The National Smelting Co., for his paper on "Discussion of the Effect of Minor Alloying Elements on Aluminum Casting Alloys," published in the ASTM BULLETIN, No. 117, August, 1942.

Mr. Crocker explained that the medal is awarded annually to the author of a technical paper considered to be of outstanding merit and constituting an original contribution on research in engineering materials. He stated that of the several excellent papers presented during the past year which represent original contributions on research in materials, the committee came to the unanimous conclusion that Mr. Bonsack's paper was both outstanding and timely. In his paper the author discussed several questions which are particularly important now because of the emergency created by the war and the important part played by aluminum alloy castings in aircraft construction and armament work. Not finding all the answers in the existing literature which he surveyed exhaustively as attested by the extensive bibliography appended to the paper, the author turned to statistical analysis of test results of several thousand melts obtained in connection with his work as Director of Laboratories at the National Smelting Co. The paper discussed in detail the effect of impurities or stray alloying elements in commercial aluminum alloy castings covered by ASTM specifications and dealt with both physical and corrosion-resistant properties, considering castability, machinability, heat treatment, and stability at elevated, subnormal, and room temperatures. Concrete conclusions were drawn as to whether various amounts of different alloying elements are beneficial or harmful, or possibly neutral, in how they affect the desired properties. It was pointed out that these conclusions should have a definite economic value in furthering a better utilization of available aluminum supplies including a more intelligent use of remelted aluminum alloys.

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[11] See p. 24.

[12] Excerpts from these addresses appear in the ASTM BULLETIN, No. 123, August, 1943.

On behalf of and as the representative of the Society, President Ball then made the seventeenth award of the Charles B. Dudley Medal to Mr. Bonsack.

In expressing appreciation of the honor conferred upon him by this award, Mr. Bonsack made the following remarks:

> I would like to express my deepest gratitude for the award of the Dudley Medal, and I would also like to thank the two committees which have done a great deal toward inspiring this paper—Committee B-6 on Die-Cast Metals and Alloys and Committee B-7 on Light Metals and Alloys, Cast and Wrought. I likewise wish to thank the National Smelting Company for putting their research facilities as well as extensive test laboratories to my use, and the only thing I can say is that I shall always cherish this medal as one of my most valued possessions. Thank you very much.

Recognition of Forty-Year Members:

The chairman announced that in accordance with action taken by the Executive Committee, certificates were being awarded this year to fourteen individuals and four companies who had attained 40 years of membership in the Society. Presentation of the certificates was then made by the Secretary-Treasurer to the following:

Frederick W. Bateman, Parker, Bateman & Chase
Almon H. Fuller, Iowa State College
Harvey G. Kittredge, The Kay and Ess Co.
J. Strother Miller, Consultant on Asphalt Technology, Rahway, N. J.
Rudolph Philip Miller, Consulting Engineer, New York, N. Y.
Leon S. Moisseiff, Consulting Engineer, New York, N. Y.
Herbert F. Moore, University of Illinois
Henry H. Quimby, Consulting Engineer, Philadelphia, Pa.
Charles Edward Skinner, formerly Asst. Director of Engineering, Westinghouse Electric and Manufacturing Co., and now retired
Maximillian Toch, Toch Brothers, Inc.
Enrique Touceda, Touceda Chemical and Physical Laboratories
C. P. Van Gundy, Baltimore, Md.
Hermann von Schrenk, Consulting Timber Engineer, St. Louis, Mo.
L. W. Walter, U. S. Maritime Commission
Carpenter Steel Co., Reading, Pa.
Arthur D. Little, Inc., Cambridge, Mass.
National Lead Co., New York, N. Y.
Pennsylvania State Highway Dept., Harrisburg, Pa.

Election of Officers:

The chairman then recognized W. A. Graham, chairman of the Committee of Tellers, who reported results of the letter ballot on election of officers. Of the 1290 ballots cast, the results were as follows:

For President, to serve for 1 yr.:
  Dean Harvey, 1290 votes.
For Vice-President, to serve for 2 yr.:
  J. R. Townsend, 1290 votes.
For members of the Executive Committee, to serve for 2 yr.:
  T. A. Boyd, 1290 votes.
  W. H. Finkeldey, 1288 votes.
  E. W. McMullen, 1290 votes.
  E. O. Rhodes, 1290 votes.
  F. G. Tatnall, 1289 votes.

The chairman then introduced the newly elected members of the Executive Committee, requesting that they rise as their names were called.

At the request of the chairman, Past-Presidents H. F. Moore and G. E. F. Lundell escorted to the chair the Vice-President-Elect, J. R. Townsend, who expressed sincere appreciation of the honor of his election. Mr. Townsend stated that he appreciated the responsibilities of the office which in these times present additional duties and problems and that he was prepared to perform the duties of the office to which he had been elected.

The chairman then requested Past Presidents Hermann von Schrenk and

H. F. Moore to escort to the chair the President-Elect, Dean Harvey, who spoke as follows in acknowledging the honor of his election:

> I appreciate more than I can say the honor which you have conferred upon me by electing me to the Presidency of the Society. I look upon it, however, not simply as an honor but as an opportunity for service. There is a very great opportunity before the Society in its war program to serve our country. You may count on me to try to do my best.

The session then adjourned following which motion pictures on "Target for Tonight" and "The Battle of Tunisia" were shown. Arrangements for showing of the films were made by the Pittsburgh District Committee through the courtesy of the Bell Telephone Co.

ELEVENTH SESSION—IRON, STEEL, METALLOGRAPHY

THURSDAY, JULY 1, 9.30 A.M.

SESSION CO-CHAIRMEN: C. F. W. RYS; E. W. UPHAM

Committee E-7 on Radiographic Testing:

Informal Report, not preprinted, presented by H. H. Lester, chairman.

Committee E-4 on Metallography:

Report presented by L. L. Wyman, chairman. In presenting the report, Mr. Wyman called upon Mrs. Beulah F. Decker to present her paper on, "Preferred Orientations in Rolled Metals: Construction of Pole Figures," to be appended to the report.

Joint A.W.S.-A.S.T.M. Committee on Filler Metal:

Report presented, in the absence of the chairman, by O. B. J. Fraser, and the following action taken:

Accepted as Tentative:

Spec. for Aluminum and Aluminum-Alloy Metal Arc-Welding Electrodes

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:

Report, not preprinted, presented in the absence of the chairman, by the chair and the following actions taken:[13]

Accepted as Tentative:

Rec. Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels

Adopted as Standard, Revisions in:

Spec. for Corrosion-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip (A 167 – 42):

Table I.—Make the following changes in the requirements as to chemical composition of the grades indicated and omit the requirements for grades 9 and 12:

Grade	Type Number	Element	Change Present Requirements From	To Read
1	301	Silicon, per cent....	0.75 max.	1.00 max.
2	302	Silicon, per cent....	0.75 max.	1.00 max.
3	304	Silicon, per cent....	0.75 max.	1.00 max.
4	308	Silicon, per cent....	0.75 max.	1.00 max.
		Chromium, per cent.	19.00 to 22.00	19.00 to 21.00
5	321	Silicon, per cent....	0.75 max.	1.00 max.
		Nickel, per cent.....	8.00 min.	8.00 to 11.00
6	347	Silicon, per cent....	0.75 max.	1.00 max
		Nickel, per cent.....	8.00 min.	9.00 to 12.00
7	302B	Silicon, per cent....	2.00 min.	2.00 to 3.00
11	316	Silicon, per cent....	0.75 max.	1.00 max.
		Molybdenum, per cent..............	2.00 to 3.00	1.75 to 2.50

Table II.—Omit the requirements as to physical properties for grades 9 and 12; omit footnote *a* and substitute therefor a reference to Section 7 (*b*).

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[13] These recommendations, not preprinted, were accepted by unanimous consent, subject to letter ballot of Committee A-10, which ballot has been favorable and the results are given in the report of Committee A-10, see p. 103.

Section 7 (b).—Change from its present form to read as follows:

(*b*) The yield strength shall be determined by the offset method as described in the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8). The limiting permanent offset shall be 0.2 per cent of the gage length of the specimen. An alternative method of determining yield strength may be used based on a total extension under load of 0.0063 in. in a 2-in. gage length.

Spec. for Corrosion-Resisting Chromium Steel Plate, Sheet, and Strip (A 176 – 42):

Table I.—Make the following changes in the requirements as to chemical composition:

Grade	Type Number	Element	Change Present Requirements From	To Read
1	403	Phosphorus, per cent..	0.030 max.	0.040 max.
		Sulfur, per cent.......	0.030 max.	0.040 max.
		Silicon, per cent......	0.75 max.	1.00 max.
		Manganese, per cent..	0.60 max.	1.00 max.
2	410	Phosphorus, per cent..	0.030 max.	0.040 max.
		Sulfur, per cent.......	0.030 max	0.040 max.
		Silicon, per cent......	0.75 max.	1.00 max.
		Manganese, per cent..	0.60 max.	1.00 max.
		Chromium, per cent...	10.00 to 14.00	11.50 to 13.50
4	430	Phosphorus, per cent..	0.030 max.	0.040 max.
		Sulfur, per cent.......	0.030 max.	0.040 max.
5	442	Phosphorus, per cent..	0.030 max.	0.040 max.
		Sulfur, per cent.......	0.030 max.	0.040 max.
6	446	Phosphorus, per cent..	0.030 max.	0.040 max
		Sulfur, per cent.......	0.030 max.	0.040 max.
		Chromium, per cent...	23.00 to 30.00	23.00 to 27.00

Spec. for High-Strength Corrosion-Resisting Chromium-Nickel Steel Sheet and Strip (A 177 – 39):

Section 4.—Change the chemical requirements for silicon from "0.75 max., per cent" to read "1.00 max., per cent."

Section 8 (b) and (c).—Substitute for these paragraphs a new Paragraph (*b*) to read the same as that recommended above in Section 7 (*b*) of A.S.T.M. Specifications A 167 – 42 except that the total extensions under load shall be as follows:

Yield Strength, min., psi.	Total Extension Under Load in 2-in. Gage Length, in.
75 000....................	0.0098
110 000....................	0.0125
135 000....................	0.0144
140 000....................	0.0148

Spec. for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (A 240 – 42):

Table I.—Make the following changes in the requirements as to chemical composition:

Grade	Type Number	Element	Change Present Requirements From	To Read
S	304	Silicon, per cent......	0.75 max.	1.00 max.
		Chromium, per cent...	18.00 min.	18.00 to 20.00
		Nickel, per cent.......	8.00 min.	8.00 to 10.00
M	316	Carbon, per cent......	0.07 max.	0.10 max.
		Silicon, per cent......	0.75 max.	1.00 max.
		Chromium, per cent...	17.00 min.	16.00 to 18.00
		Nickel, per cent.......	10.00 min.	10.00 to 14.00
		Molybdenum, per cent...............	2.00 min.	1.75 to 2.50
C	347	Silicon, per cent......	0.75 max.	1.00 max.
		Chromium, per cent...	17.00 min.	17.00 to 19.00
		Nickel, per cent.......	9.50 min.	9.00 to 12.00
T	321	Silicon, per cent......	0.75 max	1.00 max.
		Chromium, per cent ..	17.00 min.	17.00 to 19.00
		Nickel, per cent.......	9.00 min.	8.00 to 11.00

Omit the requirements for grade A, type 430 and grade B, type 446, and substitute the chemical requirements for grade A, type 410 and grade B, type 430 as given in the accompanying Table I.

TABLE I.

Grade	Type	Carbon, max., per cent	Manganese, max., per cent	Phosphorus, max., per cent	Sulfur, max., per cent	Silicon, max., per cent	Chromium per cent	Nickel max., per cent
A	410	0.15	1.00	0.04	0.04	1.00	11.50 to 13.50	0.60
B	430	0.12	1.00	0.04	0.04	1.00	14.00 to 16.00	0.60

Table II.—Omit the requirements for grade A, type 430 and grade B, type 446, and substitute the physical test requirements for grade A, type 410 and grade B, type 430 as given in the accompanying Table II.

TABLE II.

Grade	Type	Tensile Strength, min., psi.	Yield Strength min., psi.	Elongation in 2 in., min., per cent	Hardness		Cold Bend, deg.
					Brinell Number, max.	Rockwell Number, max.	
A	410	70 000	35 000	25.0	202	B88	180
B	430	70 000	35 000	22.0	202	B88	180

Committee A-7 on Malleable Iron Castings:

Report presented, in the absence of the chairman, by J. J. Kanter.

Committee A-3 on Cast Iron:

Report presented by J. W. Bolton, chairman.

Committee A-6 on Magnetic Properties:

Report presented by Thomas Spooner, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Def. of Terms, with Units and Symbols, Relating to Magnetic Testing (A 127 - 41)

Test for Magnetic Properties of Iron and Steel (A 34 - 42)

Editorial Changes Accepted in:

Test for Incremental Permeability and Core Loss of Flat-Rolled Magnetic Materials at Low Alternating Inductions Using 28-cm. Specimen (A 258 - 42 T)

Committee A-1 on Steel:

Report presented by N. L. Mochel, chairman, and the following actions taken:[14]

Accepted as Tentative, Revisions in:

Spec. for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 42 T), with the following changes:

Section 2 (a).—Change from its present form to read as follows:

2. (*a*) The steel for lap-welded pipe shall be of good weldable quality made by the open-hearth process. The steel for seamless pipe grade A, and grade B (silicon-killed), shall be killed steel made by one or more of the following processes: open-hearth or electric furnace. The steel for seamless pipe grade B (bessemer) shall be killed steel made by the deoxidized acid-bessemer process.

Section 3.—Revise the requirements as to chemical composition by changing Table I from its present form omitting the present footnote, to read as shown in the accompanying Table III.

Section 17.—Delete the words "seamless bessemer A" which appears in the first sentence in parentheses.

Adopted as Standard:

Spec. for Carbon-Steel and Alloy-Steel Ring and Disk Forgings (A 243 - 41 T)

Spec. for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 - 41 T)

TABLE III.—CHEMICAL REQUIREMENTS.

	Welded Open-Hearth	Seamless		
		Grade A	Grade B Silicon-Killed	Grade B Killed Acid-Bessemer
Carbon, max., per cent	....	0.25	0.35	0.25
Manganese, per cent	0.30 to 0.60	0.30 to 0.90	0.35 to 1.00	0.35 to 1.00
Phosphorus, max., per cent	0.045	0.04	0.04	0.11
Sulfur, max., per cent	0.06	0.06	0.06	0.06
Silicon, min., per cent	....	....	0.10	0.10

Adopted as Standard, Revisions in,

Spec. for Low Tensile Strength Carbon-Steel Plates of Structural Quality for Welding (A 78 - 40), with the following changes:

Section 3 (a).—Change the manganese content of grades A and B from "0.35 to 0.60" to read "0.35 to 0.80" per cent.

Spec. for Low Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (A 89 - 39), with the following changes

Section 3 (a).—Revise the chemical composition requirements by changing the manganese content on all four grades in Table I from "0.35 to 0.60" to read "0.80 max." per cent.

Spec. for Carbon-Silicon Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 - 39); with the following changes:

Section 1.—Change the second sentence from its present form to read as follows:

The maximum thickness of flange quality plates to be specified under these specifications shall be 2 in.; of ordinary firebox quality plates of grade A shall be 12 in.; and of grade B shall be 6 in.

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[14] These recommendations, not preprinted, were accepted by unanimous consent, subject to letter ballot of Committee A-1, which ballot has been favorable and the results are given in the report of Committee A-1, see p. 57.

Section 4.—Add under carbon, max., per cent for grade A the following:

	Grade A
For plates over 6 to 12 in., incl., in thickness	0.35

Section 8 (b).—At the end of Paragraph (*b*) add the following sentence:

The minimum elongation for grade A material shall be 15 per cent.

Section 9.—Change the table to read as follows:

Thickness of Material	Ratio of Pin Diameter to Thickness of Specimen Grade A	Grade B
1 in. and under	½	1
Over 1 to 1½ in., incl.	1	1½
Over 1½ to 3 in., incl.	1½	2
Over 3 to 4 in., incl.	2	2½
Over 4 to 6 in., incl.	2½	3
Over 6 to 9 in., incl.	3	..
Over 9 to 12 in., incl.	3½	..

Section 10 (a).—Change the second sentence from its present form to read as follows:

The fractured surface of the test specimen shall not show any single seam or cavity more than ¼ in. in length for plates ¾ in. and under in thickness; nor more than ⅜ in. in length for plates over ¾ to 4 in., inclusive, in thickness; nor more than ½ in. in length for plates 4 to 6 in., inclusive, in thickness; nor more than ¾ in. in length for plates 6 to 12 in., inclusive, in thickness, in any of the fractures obtained in accordance with Paragraph (*b*).

Section 10 (b).—At the end of Paragraph (*b*) add the following sentence:

Where the press will not break the large specimen, the specimen may be split with a gas flame or by machining, and each section broken and examined.

Table II.—Change from its present form to read as shown in the accompanying Table IV.

TABLE IV.—PERMISSIBLE VARIATIONS OVER ORDERED THICKNESS OF PLATES OVER 2 IN. IN THICKNESS.

Specified Thickness, in.	Variations over Specified Thickness for Widths Given					
	Up to 36 in., excl.	36 to 60 in., excl.	60 to 84 in., excl.	84 to 120 in., excl.	120 to 132 in., excl.	132 in. or over
Over 2 to 3, excl	1/16	3/32	7/64	⅛	⅛	9/64
3 to 4, excl	5/64	3/32	7/64	⅛	⅛	9/64
4 to 6, excl	3/32	⅛	9/64	9/64	5/32	11/64
6 to 8, excl	7/64	⅛	5/32	5/32	11/64	...
8 to 10, excl	...	11/64	3/16	3/16	...	...
10 to 12, incl	...	3/16	15/64	15/64	...	...

Editorial Changes Accepted in:

Spec. for Steel for Bridges and Buildings (A 7 – 42), with the following changes:

Section 5 (b).—Change from its present form to read as follows:

(*b*) Steel for plates and shapes 7/16 in. and under in thickness, and bars (other than those for rivets) 7/16 in. and under in thickness or diameter, intended for use in buildings and other structures subject to static loads only, may be made by the acid-bessemer process, unless otherwise specified.

Spec. for Welded and Seamless Steel Pipe (A 53 – 42), with the decimal thicknesses in Table I rounded-off to three places and Section 15 (*c*) revised as follows:

(*c*) *Couplings.*—Each length of threaded pipe shall be provided with one coupling, the threads of which shall be in accordance with the American Standard for Pipe Threads.[a] Threads shall be cut so as to make a tight joint. Couplings may be of wrought iron or steel. Taper tapped couplings shall be furnished on all weights of threaded pipe 2½ in. and larger.[b]

Spec. for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 42), with Section 13 (*c*) revised to read the same as recommended above for Section 15 (*c*) of Specifications A 53.

Spec. for Electric-Resistance-Welded Steel Pipe (A 135 – 42), with Section 14 (*c*) revised to read the same as recommended above for Section 15 (*c*) of Specifications A 53.

Spec. for Welded Alloyed Open-Hearth Iron Pipe (A 253 – 42 T). with Section 15 (*c*) revised to read the same as recommended above for Section 15 (*c*) of Specifications A 53.

Spec. for Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 – 42), with Section 14 (*c*) revised to read the same as follows:

(*c*) *Couplings.*—Each length of threaded pipe shall be provided with one taper-tapped coupling, having clean-cut threads of such a pitch diameter as to make a tight joint.

Papers:

The following papers were presented:

The Measurement of a-c. and d-c. Permeability on 28-cm. Test Specimens—J. P. Barton and G. W. Smith, presented by Mr. Barton.

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[a] A complete description of the American Standard Pipe Threads applicable to pipe, valves, and fittings is contained in the American Standard for Pipe Threads (ASA No.: B2.1-1942); also "Screw-Thread Standards for Federal Services, 1942," National Bureau of Standards *Handbook H28*, January, 1942, the pertinent data in both sources being identical.

[b] For sizes 2 in. and smaller, it is commercial practice to furnish straight-tapped couplings for standard-weight (Schedule 40) pipe and taper-tapped couplings for extra-strong (Schedule 80) and double-extra-strong pipe. If taper-tapped couplings are required for sizes 2 in. and smaller on standard weight (Schedule 40) pipe, line pipe in accordance with Specification 51 of the American Petroleum Institute should be ordered, thread lengths to be in accordance with the American Standard for Pipe Threads (ASA No. B2.1-1942). The taper-tapped couplings provided on line pipe in these sizes may be used on mill-threaded standard weight pipe of the same size.

A Test for Measuring Drawability of Deep-Drawing Steels—Francis W. Boulger and F. B. Dahle, presented by Mr. Boulger.

The Strain Aging of Killed Low-Carbon Steel, with Particular Reference to the Effect of Titanium—George F. Comstock, presented by the author.

Structure and Creep Characteristics of Cast Carbon-Molybdenum Steel at 950 F.[15]—H. E. Montgomery and John Urban, presented by J. W. Bolton.

TWELFTH SESSION—PAINT, PAPER, ELECTRICAL INSULATING MATERIALS, RUBBER, PLASTICS

THURSDAY, JULY 1, 9.30 A.M.

SESSION CO-CHAIRMEN: A. W. CARPENTER; ROBERT BURNS

Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Report presented by H. E. Smith, chairman, and the following actions taken:

Accepted as Tentative:

Test for Conducting Road Service Tests on Traffic Paints
Test for Light Sensitivity of Traffic Paints
Test for Dry-To-No-Pick-Up-Time of Traffic Paints
Test for Evaluating the Degree of Blistering of Organic Coatings When Subjected to Immersion or Other Tests Involving Exposure to Moisture or Liquids
Analysis of Barium Sulfate Pigments
Analysis of Mica Pigment
Analysis of Magnesium Silicate Pigment
Analysis of Aluminum Silicate Pigment
Analysis of Diatomaceous Silica Pigment

Accepted as Tentative, Revisions in:

Test for Phthalic Anhydride Content of Alkyd Resin Solutions (D 563 – 40 T)

Adopted as Standard:

Spec. for Chrome Yellow and Chrome Orange (D 211 – 42 T)
Spec. for Liquid Paint Driers (D 600 – 41 T), with the following revision:[16]

Section 2.—Add the following as a footnote to the table with a reference to the item for miscibility with oil:

> In case of disagreement between the seller and the purchaser, the test for oil miscibility shall be made at 25 ± 1 C.

Spec. for C. P. Toluidine Toner (D 656 – 42 T)
Testing Liquid Driers (D 564 – 41 T), with the following revision:[16]

Section 7.—Add to the description of the miscibility test in this section a note to read the same as the footnote as recommended above for Specifications D 600 – 41 T.

Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces (D 610 – 41 T)
Spec. for Dibutylphthalate (D 608 – 41 T)[16]

Adopted as Standard, Revisions in:

Spec. for Basic Carbonate White Lead (D 81 – 41)
Methods of Testing Oleoresinous Varnishes (D 154 – 38), with the following revision:[16]

Section 7.—In this section describing the test for nonvolatile matter, add the following note:

> NOTE.—A pyrex or similar heat-resistant glass Petri dish 100 mm. in diameter by 10 mm. in depth may be used in place of the flat-bottom metal dish.

Committee D-6 on Paper and Paper Products:

Report presented by G. H. Harnden, secretary, and the following actions taken:

Accepted as Tentative:

Test for Degree of Staining of Papers by Alkali
Turpentine Test for Grease Resistance of Paper with an editorial change in Section 2 (*d*) describing "book paper" in which "super calendered" is changed to read "coated."
Test for Wire and Felt Sides of Paper
Test for Surface Wettability of Paper (Angle-of-Contact Method)
Test for Kerosine Number for Roofing and Flooring Felt by the Vacuum Method
Test for Resistance of Paper to Passage of Air, with an editorial correction in Section 2 (*c*) changing "489" to read "4.89" in. of water.

Accepted as Tentative, Revisions in:

Method of Qualitative Examination of Mineral Filler and Mineral Coating of Paper (D 686 – 42 T)

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[15] Published in ASTM BULLETIN No. 125, December, 1943, p. 13.

[16] This revision, not preprinted, was accepted by unanimous consent, subject to letter ballot of Committee D-1, which ballot has been favorable and the results are given in the report of Committee D-1, see p. 256.

Adopted as Standard:

Method of Conditioning Paperboard, Fiberboard and Paperboard Containers for Testing (D 641 - 41 T)
Method of Compression Testing of Corrugated and Solid Fiber Boxes (D 642 - 41 T)
Test for Folding Endurance of Paper (D 643 - 41 T)
Test for Thickness of Paper and Paper Products (D 645 - 41 T)

It was announced that the recommendation in the preprinted report for the publication as tentative of the Test for Flammability of Treated Paper and Paperboard had been withdrawn by the committee.

Committee D-9 on Electrical Insulating Materials:

Report presented by W. A. Zinzow, secretary, and the following actions taken:

Accepted as Tentative:

Spec. for Orange Shellac and Other Lacs (D 237 - 43 T)[17]
Spec. for Low- and Medium-Voltage Pin-Type Lime-Glass Insulators

Accepted as Tentative, Revisions in:

Testing Molded Materials Used for Electrical Insulation (D 48 - 42 T)
Test for Saponification Number of Petroleum Products by Color-Indicator Titration (D 94 - 42 T), submitted jointly with Committee D-2 on Petroleum Products and Lubricants
Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 - 38 T), with a change in the new Section 39 by substituting the phrase "varnished cloths and tapes, including glass" for "varnished glass cloths and tapes."
Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 42)
Testing Laminated Tubes Used in Electrical Insulation (D 348 - 42)
Testing Laminated Round Rods Used in Electrical Insulation (D 349 - 42)
Testing Electrical Insulating Oils (D 117 - 42), with the following additional revision:[18]

Dielectric Strength.—In the revised description of the test for dielectric strength, submitted as a tentative revision[19] in June, 1940, omit Section 26 which reads as follows:

26. *Preparation of Sample.*—The sample in the container shall be shaken vigorously so as to thoroughly mix the oil and allowed to stand for 15 min. to permit the air bubbles to rise to the surface. The container shall be inverted three times before filling the test cup. The cup shall be filled with the sample to a height of not less than ¾ in. (19 mm.) above the top of the electrodes and the sample allowed to stand in the cup for 3 min. prior to the first breakdown test and 1 min. prior to the application of voltage for each succeeding breakdown.

Adopted as Standard:

Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 - 40 T)
Measuring Mica Stampings Used in Electronic Devices and Incandescent Lamps (D 652 - 41 T)

Adopted as Standard, Revisions in:

Testing Electrical Insulating Oils (D 117 - 42), with a further modification in the revised Section 1, changing "similar apparatus" to read "other electrical apparatus."
Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 42)

Editorial Changes Accepted in:

Spec. for Phenolic Laminated Sheets for Radio Applications (D 467 - 40 T)
Spec. for Round Phenolic Laminated Tubing for Radio Applications (D 616 - 41 T)

Accepted for Publication as Information Only:

Routine Test for Gas Content of Insulating Oil
Methods of Testing Askarels
Statements on Significance of Tests of Electrical Insulating Liquids

Sectional Committee C59 on Electrical Insulating Materials:

Report presented by H. L. Curtis, chairman.

Committee D-11 on Rubber Products:

Report presented by J. H. Ingmanson, chairman, and the following actions taken:

Accepted as Tentative:

Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials
Testing Compressed Asbestos Sheet Packing
Spec. for Insulated Wire and Cable: Polyvinyl Insulating Compound
Spec. for Rubber and Synthetic Rubber Compounds for Automotive and Aeronautical Applications, with revisions[20] which include the addition of another table of requirements for type R, class RS nonoil resisting compounds, also minor changes in the classification designations. In order to avoid an exces-

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[17] This recommendation was prepared jointly with Committee D-1, see Editorial Note, p. 258: also p. 333.

[18] This recommendation, not preprinted, was accepted by unanimous consent, subject to letter ballot of Committee D-9, which ballot has been favorable and the results are given in the report of Committee D-9, see p. 333.

[19] 1942 Book of A.S.T.M. Standards, Part III, p. 1592.

[20] These revisions, not preprinted, were accepted by unanimous consent, subject to letter ballot of Committee D-11, which ballot has been favorable and the results are given in the report of Committee D-11, see p. 369.

sive amount of testing, test requirements have been divided into a few basic ones and another group of optional tests; also six additional grades of synthetic rubber compounds have been added and a number of other editorial changes made in the specifications as preprinted.

Accepted as Tentative, Revisions in:

Testing Sponge Rubber Products (D 552 – 41 T)

Committee D-20 on Plastics:

Report presented by H. K. Nason, first vice-chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Vinyl Chloride-Acetate Molding Compounds

TABLE V.—ADDITIONAL DETAIL REQUIREMENTS FOR MOLDED TEST SPECIMENS.

		Type I, General Purpose				Type II, Heat Resistant		Type III, Impact Resistant				Type IV, Moisture Resistant			
		Grade MH	Grade M	Grade MS	Grade S	Grade VH	Grade H	Grade MS	Grade S	Grade VS	Grade ES	Grade VH	Grade H	Grade MH	Grade M
Heat distortion temperature, min., deg. Fahr.								125							115
Impact strength (Izod), min., ft-lb. per in. of notch	at 77 F							2.0							2.0
	at −40 F	0.4	0.4	0.4	0.4	0.3	0.3	0.5	0.5	0.5	0.5	0.4	0.4	0.4	0.4
Water absorption (24-hr. immersion), max., per cent	weight gain plus soluble matter loss							3.8							2.9
	soluble matter loss	0.5	0.7	0.9	1.1	0.3	0.3	0.8	1.0	1.2	1.5	0.2	0.3	0.5	1.0
Weight loss on heating (72 hr. at 180 F.), max., per cent		3.0	4.5	5.5	7.5	1.0	1.5	5.6	6.6	8.5	10.5	1.0	1.5	2.2	4.0
Dielectric strength (short-time test), min., v. per mil								250							

Spec. for Vinylidene Chloride Molding Compounds

Test for Shear Strength of Plastics

Method of Measuring Relative Mobility of Thermosetting Molding Powder (Cup Method)

It was announced that the recommendation in the preprinted report for the publication as tentative of the Test for Resistance of Plastics or Plastic Parts to Extreme Service Conditions had been withdrawn by the committee.

Accepted as Tentative, Revisions in:[21]

Spec. for Cellulose Acetate Molding Compounds (D 706 – 43 T), with the following revisions:

Table II.—Add the detail requirements for molded test specimens for the several properties and grades indicated in the accompanying Table V.

Adopted as Standard:

Test for Resistance of Plastics to Chemical Reagents (D 543 – 41 T)

Test for Flammability of Plastics 0.050 in. and Under in Thickness (D 568 – 41 T)

Measuring Flow Temperatures of Thermoplastic Molding Materials (D 569 – 41 T)

Test for Deformation of Plastics Under Load at Elevated Temperatures (D 621 – 41 T)

Test for Diffusion of Light by Plastics (D 636 – 41 T)

Test for Surface Irregularities of Flat Transparent Plastic Sheet (D 637 – 41 T)

It was announced that the recommendations in the preprinted report for adoption as standard of the Tests for Colorfastness of Plastics to Light (D 620 – 41 T) and for Flammability of Plastics Over 0.050 in. in Thickness (D 635–41 T) had been withdrawn by the committee.

Papers:

The following papers were presented:

Use of the Shore Durometer for Measuring the Hardness of Synthetic and Natural Rubbers—Rolla H. Taylor, presented from manuscript by the author.

Relaxation of Rubber-Like Materials—Irving L. Hopkins, presented by the author.

Deformation Under Load of Rigid Plastics—Robert Burns, presented by the author.

Impact Testing of Plastics.—I. Energy Considerations—David Telfair and Howard K. Nason, presented by Mr. Telfair.

The Relation Between Repeated Blow Impact Tests and Fatigue Tests—William N. Findley, presented by the author.

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[21] This recommendation, not preprinted, was accepted by unanimous consent, subject to letter ballot of Committee D-20, which ballot has been favorable and the results are given in the report of Committee D-20, see p. 404.

THIRTEENTH SESSION—EFFECT OF TEMPERATURE, FATIGUE

THURSDAY, JULY 1, 2 P.M.

SESSION CO-CHAIRMEN: N. L. MOCHEL; J. W. BOLTON

Committee E-8 on Nomenclature and Definitions:

Report presented by Cloyd M. Chapman chairman, and the following action taken:

Adopted as Standard:

Def. of Terms Relating to Heat Treatment of Metals (E 44 - 42 T), as revised

Joint Research Committee on Effect of Temperature on the Properties of Metals:

Report presented from manuscript by N. L. Mochel, chairman, and the following action taken:[22]

Adopted as Standard:

Rec. Practice for Short-Time Elevated Temperature Tension Tests of Metallic Materials (E 21 - 42 T)

Research Committee on Fatigue of Metals:

Report presented by H. F. Moore, chairman, who also summarized the paper by H. F. Moore and D. Morkovin entitled "Second Progress Report on the Effect of Size of Specimen on Fatigue Strength of Three Types of Steel" appended to the report.

Papers:

The following papers were presented:

The Effect of Temperature on the Transition from a Ductile to a Brittle Type of Fracture in Several Low-Alloy Steels—Paul G. Jones presented by H. F. Moore.

The Technical Cohesive Strength and Other Mechanical Properties of Metals at Low Temperatures—D. J. McAdam, Jr. and R. W. Mebs, presented by Mr. McAdam.

Interpretation of Creep-Test Data—P. G. McVetty, presented by the author.

Hyperbolic Sine Chart for Estimating Working Stresses of Alloys at Elevated Temperatures—A. Nadai and P. G. McVetty, presented by Mr. Nadai.

The Effect of Overstressing and Understressing in Fatigue—J. B. Kommers, presented by H. F. Moore.

The Fatigue Properties of Some Cold-Drawn Nickel Alloy Wires—John N. Kenyon, presented by the author.

Fatigue Tests on Some Copper Alloys in Wire Form—H. L. Burghoff and A. I. Blank, presented by Mr. Blank.

FOURTEENTH SESSION—CEMENTITIOUS MATERIALS

THURSDAY, JULY 1, 2 P.M.

SESSION CO-CHAIRMAN: P. H. BATES; J. L. MINER

Committee C-7 on Lime:

Report presented by W. C. Voss, chairman.

Committee C-1 on Cement:

Report presented by P. H. Bates, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Compressive Strength of Portland-Cement Mortars (C 109 - 37 T)

Adopted as Standard:

Test for Autoclave Expansion of Portland Cement (C 151 - 40 T), as revised

In presenting the report, Mr. Bates discussed briefly the Notes on the "Effect of Alkalies in Portland Cement on the Durability of Concrete," which appears as an appendix to the report, comprising contributions by R. F. Blanks, W. C. Hanna, Charles E. Wuerpel, F. H. Jackson, F. B. Hornibrook, H. Insley, and L. Schuman.

Papers:

The following papers were presented:

Increasing the Reflectivity of Standard Portland Cement Concretes by Additions of Hydrated Lime—C. W. Muhlenbruch and Ben Marcin, presented by Mr. Marcin.

Measurement of Bond Between Bricks and Mortar—J. C. Pearson, presented by the author.

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[22] This recommendation has been approved by letter ballot vote of the joint committee as follows: From a membership of 25, 25 members returned their ballots, 23 of whom voted affirmatively, 0 negatively, and 2 members marked their ballots "not voting."

Studies to Develop an Accelerated Test Procedure for the Detection of Adversely Reactive Cement-Aggregate Combinations—T. E. Stanton, presented from manuscript by Bailey Tremper.

A Study of the Heat of Solution Procedure for Determining the Heat of Hydration of Portland Cement—Leo Shartsis and Edwin S. Newman, presented by F. B. Hornibrook.

Variations in Strength of Portland Cements Conforming to the Same Specifications and the Relation of Such Variations to Concrete Control—S. P. Wing and Arthur Ruettgers, presented from manuscript by Mr. Wing.

FIFTEENTH SESSION—CONCRETE

THURSDAY, JULY 1, 8 P.M.

SESSION CO-CHAIRMAN: ALEXANDER FOSTER, JR.; A. R. ELLIS

Committee C-8 on Refractories:

Report presented by S. M. Phelps, secretary.

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by F. H. Jackson, chairman, and the following action taken:[23]

Adopted as Standard:

Test for Volume Change of Cement Mortar and Concrete (C 157 - 40 T), revised as follows:

Section 2.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> 2. (*a*) Molds for Mortar *and Concrete* Specimens.—Molds for casting *the* prismatic test specimens [of cement mortar] shall be made of metal and so [constructed] *designed* that the specimen [may] *will* be cast *with its longitudinal axis* in a horizontal position. The molds shall be fitted with metal end plates that provide means for holding a stainless steel [contact] *gage* point (Note 1) in exact position at the center of each end of the specimen during the placing and hardening of the *cement mortar or* concrete. Details of a suitable mold (*designed for mortar specimens*) are shown in Fig. 1.
>
> NOTE 1.—The device for holding the contact points shall be arranged so that it can be *partially* released [slightly] after the mixture has been compacted into place in order to avoid restraint of the contact points during initial shrinkage of the specimen.

Delete the present Paragraph (*b*) and reletter the subsequent paragraph accordingly.

Figure 2.—Delete Fig. 2 showing the mold for horizontal casting of cylindrical concrete specimens and renumber the subsequent figure accordingly, and change the reference in Note 2 to read "Fig. 2" instead of "Fig. 3."

Section 3.—Change to read as follows by the addition of italicized words and the omission of the words in brackets:

> 3. (*a*) Mortar Specimens.—The test specimen for cement mortar shall be a prism 2 in. square and 11 in. in length [and shall be cast with the longitudinal axis of the prism in a horizontal position].
>
> (*b*) Concrete Specimens.—The test specimen for concrete shall be a [cylinder] *prism* 3 in. [in diameter] *square* and 11 in. in length. [The maximum size of aggregate in the concrete for such a specimen shall not exceed ¾ in. If desired, the cylindrical concrete specimens may be cast with the longitudinal axis of the cylinder in a horizontal position, provided a mold of suitable design is used.]
>
> (*c*) Contact *or* Gage Points.—Each test specimen shall have embedded at each end of the longitudinal axis, a stainless steel contact *or gage* point [or surface] so designed as to provide an effective [shrinkage] *gage* length of 10 ± 0.01 in.

Section 4 (*b*).—Change from its present form: namely,

> (*b*) All materials shall be measured and proportioned by weight. The measuring and mixing of the materials and the placing of the concrete in the molds shall be performed as described in the Standard Method of Test for Compressive Strength of Concrete (A.S.T.M. Designation: C 39) of the American Society for Testing Materials. Specimens of cement mortar shall be molded by the same procedure as prescribed for concrete specimens whenever possible.

to read as follows:

> (*b*) All materials shall be proportioned by weight. The mixing and molding procedure shall follow, as nearly as possible, those prescribed in the Standard Method of Test for Flexural Strength of Concrete (A.S.T.M. Designation: C 78) of the American Society for Testing Materials.

Section 5.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> 5. (*a*) Immediately after molding, the test specimens in the molds shall be covered with wet burlap or placed in the *moist* [storage] room. After 20 to 24 hr. the specimens shall be removed from the molds and placed in the storage room.
>
> NOTE.—In the case of certain slow-hardening cements it may be necessary to allow specimens to remain in the mold for more than 24 hr. in order to avoid damage during removal from the molds. When making tests in which such cements are included, the storage schedule may be modified, but *any* [all] specimens involved in the test program, *which are to be used for comparison*, shall be subjected to identical conditions of storage.
>
> (*b*) The storage room shall be maintained at a temperature of *70 ± 2 F*. [80 ± 2 F.] and a relative humidity of 50 ± 5 per cent.

Papers:

The following papers were presented:

Simplified Test for Evaluating the Effectiveness of Concrete Mixers—S. P. Wing, Valens

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[23] This recommendation, not preprinted, was accepted by unanimous consent, subject to letter ballot of Committee C-9, which ballot has been favorable and the results are given in the report of Committee C-9, see p. 246.

Jones, and R. E. Kennedy, presented by Mr. Wing.

Effect of Curing Methods Upon the Durability of Concrete as Measured by Changes in the Dynamic Modulus of Elasticity—Bartlett G. Long and H. J. Kurtz, presented from manuscript by Mr. Long.

A New Aspect of Creep in Concrete and Its Application to Design—Douglas McHenry, presented by title.

Effect of an Air-Entraining Admixture on the Resistance to Freezing and Thawing of Concrete Containing Inferior Coarse Aggregate—E. O. Axon, T. F. Willis, and F. V. Regael, presented from manuscript by Mr. Axon.

Strength of Concrete as Related to Abrasion of the Blast-Furnaced Slag Used as Coarse Aggregate—Fred Hubbard and H. T. Williams, presented by Mr. Hubbard.

There being no further business, the Forty-sixth Annual Meeting adjourned *sine die.*

August, 1943.

THE RELATION OF THE A.S.T.M. TO THE TEXTILE INDUSTRY

ANNUAL ADDRESS BY THE PRESIDENT

HERBERT J. BALL[1]

JUNE 30, 1943

The topic which I have chosen for this address is "The Relation of the American Society for Testing Materials to the Textile Industry." It is my intention to set forth the important benefits which I believe have accrued to the textile industry during the twenty-nine years it has been served by the A.S.T.M. This means that I shall present to you a concise picture of the accomplishments of the standing committee of the Society known as Committee D-13 on Textile Materials, for it is through this committee, chiefly, that the A.S.T.M. has had its influence upon the textile industry. Do not be surprised if, in certain Society matters, my description bears a close resemblance and seems equally applicable to the standing committees of which you are members, for in reality Committee D-13's picture is very typical of many other committees in the A.S.T.M. organization.

I shall assume that my audience is composed largely of non-textile men. In order to give you an appropriate background against which to view the textile activities of the A.S.T.M., I shall quickly sketch some of the features which I believe are characteristic of the textile picture.

Let me begin by renewing your acquaintance with fibers, the fundamental raw materials of all textile products. They fall into two major classifications, natural and man-made. The important natural fibers are cotton, wool, silk, asbestos, alpaca, camel's hair, cashmere, mohair, vicuna, and those in the bast and leaf group of which flax, jute, hemp, ramie, and sisal are probably better known to you. Today a list of the commercial man-made fibers would include acetate rayon, cuprammonium rayon, viscose rayon, nylon, glass, and others derived from various resins, casein, and the soybean. What new fibers the postwar tomorrow will bring I will not attempt to predict, but that it will bring new and valuable developments in this field is undoubted.

Just a thought or two for those who are inclined to look askance at textiles as an engineering material, or who think of textiles merely in terms of wearing apparel, curtains, upholstery, sheets or other articles of common or personal use. A few illustrations of the so-called mechanical uses may be helpful at this time in making clear how textiles are engineering materials in their own right or as essential components of others: belts for transmission of power, conveyor belts, fire hose, filter cloths, brake bands, sails, tentage, thread, rope, printers' press blankets, paper-makers' felts, balloons, parachutes, parachute webbing, covering of airplane wings and bodies, gas masks, tire cords, and tire fabrics. I shall cite other important uses later.

To emphasize this point further, I turn your attention to the man-made fibers previously mentioned. Here we have textile materials whose chemical and physical properties are largely determined by the producer before manufacture. In the same manner that the properties of steel are varied by changes in its chemical composition and method of production, so the producer of rayon, for example, can alter its strength, elongation, moisture content and other fundamental qualities. Fibers which are thus under the control of man's engineering genius are certainly to be classed as engineering materials, and, like their near relative, plastics, are worthy of engineering consideration alongside of steel, concrete, and other construction materials.

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[1] Professor of Textile Engineering, Lowell Textile Institute, Lowell, Mass.

Some comments upon certain features of the textile industry as a whole are next in order. The entire textile industry which the A.S.T.M. serves is actually composed of many separate industries. The recognized branches or subdivisions of it are differentiated according to the textile fiber with which the group chiefly deals. Thus, for example, we commonly refer to the cotton industry, to the wool industry, or to the rope and cordage industry. The subdivisions of the textile industry are very closely analagous to the subdivisions of the ferrous metals field for which the A.S.T.M. has set up separate standing committees under the titles of A-1 on Steel, A-2 on Wrought Iron, A-3 on Cast Iron and so on. In the case of textiles, it should be noted that one standing committee, D-13, serves the entire field.

Without entering into the details of the organization of Committee D-13, it will suffice to say that it has been developed in such a manner as to provide for every important fiber branch of the industry, for the important subdivisions within each branch, and for those functions which are of general applicability to all branches, that is, to the industry as a whole.

The textile industry has long been a traditional one. Only in comparatively recent years has the value of research and of the scientific approach to the problems of manufacturing and merchandising been generally realized by a substantial part of the industry. This changed attitude has resulted in the increased employment of technically trained personnel, and, in very large measure, these are the men and women who have been actively engaged in advancing the standardization activities with which we are presently concerned. It can be said that the industry is now emerging from the doldrums of tradition and taking its place with other progressive industries.

A textile man will recognize that the foregoing is far from a complete discussion of each topic. For the non-textile person, however, I trust it has been sufficiently adequate to provide a suitable background against which to view the general standardization problems of the industry and to make for a better understanding of what has been done in this field.

How widely interest in the standardization program has spread to the textile and to associated industries can be determined in part by a scrutiny of the business connections of those engaged in the work. It will be found that in each fiber branch there are representatives from all the larger producers of yarns and fabrics, and in most instances from their respective national associations. The producer group also includes the names of all units of the chemical industry which produce rayon fiber. Consumer interests are represented by large mail-order and chain store organizations, large retail stores, wholesalers and distributors, a national purchasing organization, consumer advisory groups, well-known tire and rubber companies, manufacturers of electrical equipment, wire manufacturers, and railroads. The general interest group includes those from government departments and bureaus of the United States and Canada, commercial testing organizations, colleges, technical schools, builders of testing equipment, and manufacturers of soaps, dyestuffs, and finishing agents.

One direct measure of the magnitude of the standardization activities is to be found in the 78 textile standards that are now on the books of the A.S.T.M. The indirect and equally valuable effects of the years of work, meetings, interlaboratory tests, and discussions are difficult of reduction to a common denominator, but that they have been very great and influential will hardly be denied.

I do not propose to bore you with a lengthy examination of each of these standards but, on the contrary, will cite such illustrations as will make clear their importance, utility, or the progress they denote. In 1914, when the A.S.T.M. authorized the formation of the textile committee, methods of test were in a chaotic condition. This was particularly true, for example, of the breaking strength test where size of test specimen, type of clamps, and speed of pulling clamp differed with the individual thinking at any one mill. Furthermore, the mills tested their products under the atmospheric conditions prevailing in the spinning or weaving rooms, office or basement, wherever the testing equipment happened to be located. The standardization of this test and its technique was obviously one of the first requisites if comparable test results between producer and consumer were to be obtained. The results of the studies made at that time have come down to us today in the widely accepted standard requirements for the grab and raveled strip test specimens, for standard atmospheric conditions for testing laboratories, and for strength testing machines and the details of their operation.

Other standards, developed later, and also of general applicability to and acceptance by the entire industry are those for bursting strength testers, for twist testers, for the dead weight gage for thickness measurements, the method of determination of twist in yarns, of identification of fibers in mixtures, of color fastness of colored textiles to light, the fundamental textile definitions, and the **S** and **Z** nomenclature for designation of direction of twist. This latter item has in fact become an international standard through adoption by the International Standards Association Committee 38 on Textiles. The abovementioned standards alone have unquestionably had a marked influence upon the manufacturing, sales and testing activities of every important producer and consumer in the entire textile field.

Each separate branch of the industry has naturally felt the influence of the particular standards prepared for its own needs by its own representatives. In evaluating this effect, it must be remembered that the cotton industry is the largest of all. Its annual output is measured in billions of pounds and on this basis is larger than all the other fiber groups combined. This industry has been provided with methods of test for cotton in all its important forms, namely, fiber, yarn, thread, the many mechanical fabrics ranging in weight from light to heavy, and those fabrics which enter so largely into the ultimate consumer field. With the cooperation of the U. S. Department of Agriculture, a set of appearance standards has been developed for the purpose of grading cotton yarns for certain qualities which can be determined by visual examination. These have been found to be very useful and have been well-accepted.

The rubber tire industry was the first and has always been one of the most active in the promulgation of standards for cotton textiles and more recently for rayons. This work has so far advanced that tire textiles are now produced to meet rigid specifications for strength, elongation, twist, weight, and thickness. Inadequacies in these properties are directly reflected in the quality of the tires produced from them, and ultimately in the safety of those who ride on them. This industry quite generally writes into its purchase specifications the A.S.T.M. methods of test, and it has been indicated that the Society's material specifications for tire textiles have resulted in a reduction in the variety of fabrics used in the industry.

The woolen and worsted industry had practically no technical standards until they were supplied through the activity of the wool group of Committee D-13, beginning about twelve years ago. The energy and diligence of this group has now given the industry standard methods of test for the material in the form of fiber, woolen yarns, worsted yarns, mixture yarns, wool and part wool fabrics, and felt. The test for fineness of wool tops has substituted a scientificprocedure for one in which the personal equation previously predominated, has resulted in the setting of grade standards, and has given the industry a tool of great value in settling fairly disputes with government agencies about the grades of grease or scoured wools. The test has been made the basis for the grading of tops by the Wool Associates of the New York Cotton Exchange, has received official recognition by the U. S. Department of Agriculture, and is employed by the Army and Navy to determine the grade of wool in fabrics.

Working in close cooperation with the U. S. Treasury Department, there has been developed, as an A.S.T.M. standard, a new method of estimating hard scoured wool in wool in the grease. This represents a solution of an old and difficult problem of determining wool shrinkage in connection with tariffs and duties. The method is now daily routine work at the U. S. Customs Laboratory in Boston. As another illustration of cooperation, I shall cite the present work on the establishment of grades for the coarser wools. The groups represented are the Boston wool trade, the Philadelphia wool trade, the carpet industry, the felt industry, the National Association of Wool Manufacturers, the U. S. Department of Agriculture, and the U. S. Treasury Department. It is only natural that A.S.T.M. standards developed by such cooperative approach should be highly authoritative in the industry concerned.

The requirements of the automotive industry as large buyers of wool felt have stimulated interest in this material by the wool group. The result is a carefully drawn set of physical and chemical test methods for this important textile.

Members from the rug and carpet industry have provided it with a comprehensive set of methods of test of pile floor covering which is now the guide of that industry. This has come about because its research and testing engineers could meet in the technical atmosphere of Committee D-13 to operate on matters affecting the welfare of the industry as a whole, without having to consider personal and competitive conditions. Under such auspices the units of this industry have found it possible to unite in an extensive wear test program, in cooperation with the National Bureau of Standards, from the results of which it is hoped to standardize on the ultimate wear of pile floor coverings.

Standardization activity in the rug and carpet field has spread to the jute industry because the latter is the supplier of one of the important materials entering into the construction of floor coverings. The representatives of the jute industry have set up referee methods of test for four important types of its products whereby buyer and seller can refer disputed deliveries to a neutral competent technician, and decision may be reached by comparing test results with purchase agreements. The inclusion of these methods in the purchase specifications of nonmembers is not only gratifying but indicates that the standards are filling a need.

The glass fiber industry, the latest group to join in the activities of the Textile Committee, has already developed standard methods of test and tolerances for glass fabrics, tapes, sleeving and yarn, together with definitions of pertinent terms. It has had the advantages which come from the initiation of standardization work soon after glass fiber products were in commercial production. There was also available to it all of the fundamentals regarding testing conditions and machines which had been developed for other textile products. These were incorporated with little or no change for testing glass textiles.

Manufacturers of electrical equipment are important consumers of quantities of textile materials for insulating purposes. These take the forms of roving, yarn, sheeting, duck, tape, tubular sleeving, and braid and are made of cotton, rayon, silk, jute, asbestos or glass. Representatives of the producers and consumers have worked out satisfactory methods of test for each of the above textile products to determine their suitability for insulating electrical conductors. These standards have been of particular benefit to the asbestos industry. Prior to their adoption, individual manufacturers of electrical apparatus had separate specifications and methods of test which not only differed but which placed a hardship on the supplier by requiring rather complete and unnecessarily burdensome test apparatus.

The rayon industry has developed all of its important standardization work under A.S.T.M. auspices. Three standards cover the entire rayon yarn field, one dealing with filament rayon, one with rayon staple, and the other with yarns and threads made from the staple fiber. The extent to which the effect of the methods of test found in these standards permeates the whole textile industry will be better conceived when one recalls the phenomenal expansion which has occurred in the past fifteen to twenty years in the use of this material and which has made it the second largest branch of the textile industry measured on the basis of poundage output. Cotton, woolen, worsted, and silk manufacturers, as well as the knit goods industry, are consumers of enormous quantities of rayon to be turned into yarns and fabrics made wholly of rayon or in combination with the other fibers. The advantages which come from having the various fiber groups working under a common banner is further evidenced in the rayon staple fiber field. It is found that the methods developed by the cotton and wool groups for the sampling and determination of length of cotton and wool fibers, respectively, are quite adaptable to the various class lengths of rayon staple. It should be noted that the Federal Trade Commission in its rules for the rayon industry has adopted the A.S.T.M. definition of rayon. The rayon manufacturers, weavers, and government agencies now regularly employ the rayon standards in the development and inspection of rayon fabrics for ordnance and aircraft use, particularly in high-strength automobile, truck, and aircraft tires, and in balloons and parachutes.

Large quantities of fabrics are finally

converted into a wide variety of articles to satisfy the needs and desires of the so-called ultimate consumer. Interest in textile commodities of this type has shown a rapid increase in recent years as a result of the educational work, particularly among women, by the departments of home economics in the colleges, by state and national bureaus, and by various consumer organizations. This demand for more information about textiles is ultimately reflected in statements on tags or labels regarding such properties as strength, weight, construction, color fastness, and fiber content. Determination or verification of such facts raises the question of reliable test methods and specifications and this is work for the A.S.T.M. In recognition of this, Committee D-13 has organized a group whose function is the preparation of standards for household and garment fabrics. It has done this in the belief that this offers to the textile industry and to those who are closest to the consuming public the most logical meeting place wherein to work out the best compromise standards, practical in their requirements and fair alike to both interests. The results to date are tentative specifications for five fabrics, namely, broadcloth, sheeting, terry toweling, corduroy, and blanketing. It will be noted that these deal with the material out of which the ultimate consumer goods are made. It is not the intention to deal with the final articles themselves.

The Textile Committee has realized the need for a better understanding of the relations between sampling procedures and the proper number of tests it should specify in its standards. The group which it organized to take over the problems connected with sampling and interpretation of data is doing a most excellent piece of educational work. As a result there has already been set up a definite procedure for the use of the subcommittees when it is desired to specify the number of tests necessary to determine the average quality of a lot or shipment of a textile material with respect to an individual quality characteristic. It is also to be used when it is desired to fix the sample size on the basis of an allowable error of the average and for a chosen probability. It is adaptable for use by individual manufacturers and consumers in developing company standards or specifications. It is not surprising, therefore, that a textile standard is the first Society standard to have incorporated in it a statement regarding the expected precision of a test result. The entire program has undoubtedly been largely instrumental in making the textile industry conscious of the need for and value of statistical control.

Other benefits have accrued besides those industry-wide ones previously mentioned, and the committee's standards have been used to advantage in many ways by individual units in the industry. Disagreements between buyer and seller, and customers' complaints as to quality are frequently settled by use of the A.S.T.M. textile standards in recognition of their fair, reliable, unbiased, and authoritative character. Definite reference to them in purchase contracts has tended to preclude controversy and litigation, and to eliminate losses and delays through possible misunderstandings and misinterpretations.

The standards have served as guide posts to the commercial testing organizations in fulfilling the many and varied requirements their work entails. The compilation of textile standards, a book containing all the Society's standards relating to textile materials, is found to be a handy ready reference manual in most textile testing laboratories, and is used extensively as the text book in classes in textile testing in the schools and colleges. By the committee's work, testing machine builders have been stimulated to develop new and better pieces of testing apparatus, and the fact that a piece conforms to an A.S.T.M. requirement is regarded as a mark of approval and distinction. Mill research laboratories have recognized that the textile standards may be employed as an effective means for securing reliable processing control. Some smaller units have been spared the expense of developing laboratory methods of their own and have eliminated much time-consuming controversy within the four walls of their plant by reference to the standards. The use of standard test methods between mills, brokers, selling houses, and the consumer assures a better comparability of test results as the properties of a material are evaluated by each interested handler along the channel of distribution.

The meetings of Committee D-13 have become one of the most important clearing houses for the exchange of technical information of value to the industry as a whole. Producer, consumer, and general interest members seem very willing to make their contribution to the advancement of the knowledge of textile materials and their properties. Papers presented before the committee as a whole and at the meetings of its subgroups offer the very newest in facts, techniques, and developments. This has permitted a coordination, between the divisions of the industry, of the attack on problems of related interest, and has undoubtedly been effective in speeding up research work in individual companies. Into these papers and resulting discussions are poured the great wealth of accumulated experience and new knowledge possessed by representatives of government bureaus and departments, by experts and technicians in the research, developmental, and testing laboratories of large industrial plants and consumer establishments, and by those inspired to research in textiles in the schools and colleges throughout the country. The wide representation of interests and the substantial body of scientific men and women which are brought together at its meetings affords an unexcelled opportunity to broaden one's knowledge, and the contacts made with the minds of others lead to intellectual growth and professional maturity.

It is very clear that the textile industry recognizes in the A.S.T.M. a central organization, covering the entire fiber field, and doing practical, scientific standardization work of the broadest scope. The continuous increase in members from this industry is excellent evidence of the Society's importance to the industry which it serves. Under its auspices producers and consumers meet on neutral ground and in a technical atmosphere, acquire a mutual respect for the other's difficulties, and then cooperate in the solution of their common problems. It is highly essential to the industry that those who are most experienced in and who have intimate contact with the standardization work of their respective organizations should serve in the development of the Society's standards. To this end, the A.S.T.M. invites the continued support of the industry through membership of its qualified men and women.

The quality of the standards which the textile industry has prepared for itself, coupled with the prestige and the sensible requirements of the A.S.T.M., has placed them in an authoritative position in the industry. This position must not be impaired one iota. Those who put faith in the integrity and scientific character of our work must not have that confidence shaken or violated. To see that the standards shall be reliable, unbiased, practical, technically correct, and comprehensive, must be the standard upon which the eyes of its officers and committee chairmen are constantly focused. Under such conditions the textile industry will continue to accept the leadership of the A.S.T.M. in the standardization field, and its influence will become even more widespread and effective as the years roll by.

ANNUAL REPORT OF THE EXECUTIVE COMMITTEE

Of first importance in this annual report of the Executive Committee to the Society is an accounting of the activities of the Society in relation to the war. In an "all-out" war such as our country is now waging, the performance of all portions of the body politic—technical societies as well as industries, companies, and indeed, individuals—must periodically be measured with the yardstick of value to the war effort. Not that this is the sole measure of worth; the ideals and principles upon which our nation is founded must be preserved even during the stress of war, and this calls for determined effort to carry on important things in as nearly a normal way as possible. Thus, the Executive Committee has deemed it essential to give the usual careful thought to the planning and carrying out of the fundamental things in the Society—sound growth in our membership; efficient committee organization and procedure; maintenance of essential research and standardization in materials and specifications; publication of technical data, through reports, papers, and standards; national and district meetings; effective cooperative relations with other technical and industrial organizations and with Governmental agencies; and assurance of adequate financial support for all of these activities. How well these things are being done may, we believe, be gathered from the pages of this report, from the news of Society affairs in the ASTM BULLETIN, from the technical publications, and from the annual meeting at which this report will be presented. But, until the war is won and the security of our nation assured, the most important measure of our accomplishments is their contribution to the winning of the war. And, in this introductory portion of its report, the Executive Committee examines some of these accomplishments in that light.

The basic war-time policy of A.S.T.M. was established soon after Pearl Harbor: in brief, to make available all of the Society's facilities to assist the Government and industry in dealing with any problems within the Society's traditional field of engineering materials. Because this is basically a war of materials and of resources for production of the implements of war,—the familiar "planes, guns, tanks and ships,"—the activities of the Society and the experience of its members and committees in research and standardization of materials have provided many opportunities for contributions toward the problems of specifications and procurement of materials and of expediting war production. One of the first steps taken as we entered first the period of defense and then of war emergency was to provide certain modifications in normal standardization procedure and more particularly to develop special procedures for handling emergency revisions in specifications and for issuing entirely new emergency specifications, in order that the Society could keep well abreast of the constantly changing situation with respect to the supply of critical materials and provide "up to the minute" specifications for most of the important materials of war. These procedures make use of action by Committee E-10 on Standards on all committee recommendations for emergency standards without requiring action by the Society in annual meeting or letter ballot, thus greatly speeding the issuance of emergency standards, which, of course, will be subject to usual Society action if later on any of them are proposed for permanent adoption.

By these procedures there have been issued a total of 106 emergency alternate provisions covering principally the ferrous and non-ferrous metals but including also a number dealing with cement, asphalt roofing, rubber products, and detergents. There are also 31 new emergency specifications, many of which are playing an important rôle in war production. While many of these emergency alternate provisions and specifications have been developed on the initiative of our committees, many others have been written at the direct request of, or to meet needs expressed by, some agency of the Government such as the Army, Navy, and particularly the War Production Board. The specifications in the latter category include a majority of the 95 items dealing with metals. In addition to this emergency specification work the committees have also developed many new tentative standards, which were pushed to completion for inclusion in the 1942 Book of A.S.T.M. Standards.

The 1942 Book of Standards itself is one of the Society's notable contributions. Extensive committee work prior to the last annual meeting enabled the Society to bring out the most complete compilation of materials standards that it has ever published, comprising 688 standards and 382 tentative standards, in addition to nearly all of the emergency alternate provisions and emergency specifications referred to above. Despite publication difficulties occasioned by war conditions, this largest volume of its kind was brought out in three parts early this year and is serving war needs throughout the nation. But the work of developing new standards has gone right on; since this volume was published there have been approved and published some 10 new emergency specifications and 18 new tentative standards. Moreover the committee work in the past year culminating in reports to be considered at this annual meeting and by Committee E-10 on Standards this summer will result in additional new standards and revisions of existing standards for publication in the 1943 Supplements to Book of A.S.T.M. Standards this fall.

To an increasing extent, the standards of the Society, especially the emergency revisions thereof, are being used officially by the Government by reference thereto in limitation and material orders of the War Production Board and as the basis for price regulation or in conjunction therewith by the Office of Price Administration. The most important instance of this kind is in the War Production Board Limitation Order L 211 covering National Emergency Specifications for Steel Products, in the various schedules of which numerous A.S.T.M. specifications for steel and steel products are specified to govern the production and delivery of those products during the war emergency. In this work, which has been carried on in cooperation with the Society of Automotive Engineers, American Iron and Steel Institute, and the War and Navy Departments, the Society has utilized its facilities very effectively in furtherance of war production. Issues of the ASTM BULLETIN currently record the progress of this work, and this year's Report of Committee A-1 on Steel makes appropriate reference to it. That committee and its members have done a great deal of valuable work in the technical advisory committees of National

Emergency Steel Specifications jointly sponsored by the bodies above named. Much work still lies ahead of the Society in this field.

Other references to A.S.T.M. specifications are found in WPB Limitation Orders L 179 on cement, L 252 involving valves and flange parts, L 278 on steel pipe fittings, and other orders refer to various A.S.T.M. methods of tests.

Mention should also be made of cooperation with Federal specification committees and with specification groups in various bureaus of the War and Navy Departments. Some of the fields in which such cooperative work has been carried on include malleable iron flanges, pipe fittings, and valve parts; fire-refined copper for wrought products and alloys; copper-base alloy castings, including further reduction in the number of compositions for casting alloys; special quality of zinc-base and aluminum-base die castings; soaps and detergents; specifications for natural and synthetic rubbers, developed in cooperation with the Society of Automotive Engineers under the auspices of Committee D-11; and various plastic compounds, to which further reference is made below.

So much important work on standards has been carried on in the committees of the Society for report at this annual meeting that it cannot be presented in any brief summary here. There is, however, one outstanding accomplishment that deserves special mention and that is the work being done by Committee D-20 on Plastics in the development of specifications for the principal plastic molding compounds and for sheets, rods, tubes, and shapes made from the various plastics. Committee D-9 on Electrical Insulating Materials together with Committee D-20 have developed extensive methods of test for plastics and the former committee has contributed a number of specifications applying particularly to the use of plastics in the field of electrical insulation. After several months of intensive work Committee D-20 submitted ten tentative specifications for plastics which although approved too late for inclusion in the 1942 Book of A.S.T.M. Standards have been published in the compilation of A.S.T.M. Standards on Plastics issued in May, 1943. The committee is also completing other specifications including some covering the nonrigid variety of plastics that are of special interest in the automotive and aircraft fields. There has been a great need for authoritative standards for plastics in view of very extensive use in many phases of war production, and the work of these committees has enabled the Society to make an important and timely contribution to the war effort.

The Society has used every means at its command and has spared no expense to make its standards and other technical publications available in the most convenient form. Thus, as noted in detail in a later section of this report, 13 compilations of standards in various fields have been issued, for which there has been an extensive demand. These compilations distribute A.S.T.M. standards very broadly throughout industry thus bringing them more widely to the attention of engineers, designers, and specification writers and making them more readily available for application to war production. The utility of these and many other of the Society's accomplishments is a direct result of publication policies established years ago in an effort to make the Society's standards readily available.

What has been said so far deals primarily with the value of the Society's output—standards, reports and other publications—to the prosecution of the war, and the Executive Committee feels that the record is one in which the Society can take much pride. However, no reference to the Society's contribution would be complete without mention of the fact that many members of the Society are serving either in the Armed Forces, in some agency of the Government or in industry in important and key capacities. Committees of the Society have on frequent occasion given advice and comment on materials problems and some have carried on special studies to ascertain facts and determine information that will be of help. The Society has responded to many requests for information and copies of publications.

During the past year the Secretary-Treasurer has continued his services in the War Production Board, where he is Chief of the Materials Branch of the Conservation Division. About three fourths of his time is devoted to this work.

A number of the items referred to above are reflected in the committee reports being presented at this annual meeting and these, together with a number of the technical papers which also represent direct contributions to the war effort give the annual meeting a decided war atmosphere. This, of course, is in line with the policies established by the Society that all of its work should be reviewed in the light of its contribution to the war effort and it is but natural that so many of the items on the program would partake of this nature. This is further reflected in the fact that most emphasis at this annual meeting is being placed upon the holding of committee meetings since many of these committees have important problems before them that will require their attention.

The emphasis on war contributions, however, is not to the exclusion of committee work and papers that have a long-range significance and which because of their intrinsic importance in the promotion of knowledge of materials should be continued in the interest of maintaining this work on a sound basis for the post-war period.

Committee Activities:

The current Reports of Committees E-9 on Research and E-10 on Standards deal with certain phases of the committee work and, supplemented by reviews that will appear in the ASTM BULLETIN, give a general summary of the committees' activities. This report, therefore, makes brief reference only to several administrative actions during the year.

The Executive Committee has had before it several jurisdictional questions. One of the most important of these relates to the clarification of the relations between Committees D-4 on Road and Paving Materials and D-18 on Soils for Engineering Purposes, with respect to development of methods of testing and specifications for stabilized soils. A plan for cooperation between the two committees suggested in the report of the Executive Committee two years ago[1] was found to require further study in the light of representations made by members of the two committees. Accordingly a conference committee consisting of three representatives from each of the two commitees, chairmaned by Vice-President Harvey, has unanimously agreed upon a revised plan of cooperation which has met with the approval of the Executive Committee and has been submitted to the two committees to be put into effect. In brief, this plan contemplates a single joint subcommittee on tests and specifications for stabilized soils, which shall be responsible to both committees and function under the joint direction of the advisory committees of Committees D-4 and D-18. When this joint subcommittee is organized, the present Subcommittee D-5 of Committee D-4 and Subcommittee V of Committee D-18 will be discharged. It has further been suggested to Committee D-18 that the committee may wish to review its scope with a view to including work on specifications for soils.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 49 (1941).

Another jurisdictional matter had to do with the Tentative Method of Salt Spray Testing of Non-Ferrous Metals (B 117 – 41 T). As mentioned in the report last year,[2] Committee E-10 on Standards had recommended that jurisdiction for this method be transferred from Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys to Committee E-1 on Methods of Testing, inasmuch as the expanded scope of the method makes it of interest to several other committees. Consideration by the Executive Committee of discussions of this matter during the year in the Advisory Committee on Corrosion has led to the decision to continue jurisdiction in the hands of Committee B-3, with the understanding that interested groups should be kept in touch with any developments regarding the method. It is the feeling of the Advisory Committee that work in the present corrosion committees can be adequately coordinated through the Advisory Committee and that Committee E-1 on Methods of Testing should not be asked to initiate work in the corrosion fields.

Discussion of jurisdictional questions between Committees D-11 on Rubber Products and D-20 on Plastics has resulted in an agreement between the two committees establishing an arbitrary line of demarcation in jurisdiction over rubber-like materials which might be classified either as synthetic rubbers or as plastics. Accordingly the following sentence was added to the statement of scope of Committee D-11 as printed in the 1942 Year Book:

> "Rubber-like materials referred to in this scope comprise butadiene synthetic rubbers (Buna, Chemigum, Hycar, Perbunan); butyl rubber; Koroseal; neoprene; polyisobutylene; Thiokol; and latices from any of the above materials."

The very general substitution of synthetic materials for natural rubber and the difficulties in establishing lines of demarcation between plastics and synthetic rubbers have led to reopening this jurisdictional question, and the two committees are now studying redefinition of their respective scopes.

The following change in scope of Committee D-19 on Water for Industrial Uses has been approved by the Executive Committee at the request of Committee D-19, the new material appearing in italics:

> "The study *and description* of water as an engineering material for industrial purposes. *This will include* terminology; methods of sampling analysis *and testing*; interpretation of results of *analyses and* tests; classification; and *the preparation of* specifications."

Inquiry was made of Committee E-7 on Radiographic Testing regarding the need for the classification of its personnel as to producers, consumers, and general interests, since it is the only one of the testing committees to be so classified. Committee E-7 has advised that since it will at times prepare standards of a commercial nature it wishes the classification continued, and the Executive Committee has agreed to allow the classification to stand.

On the recommendation of the Advisory Committee on Corrosion, the Executive Committee has extended invitations to Committees B-7 on Light Metals and Alloys, D-1 on Paint, Varnish, Lacquer, and Related Products, and

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[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 55 (1942).

D-19 on Water for Industrial Uses, to name representatives to the Advisory Committee.

The following two joint committees have been discontinued for the reasons given: (1) Joint Committee on Standard Specifications for Concrete and Reinforced Concrete has completed its work; and (2) Coordinating Committee on Weathering Characteristics of Masonry Materials, consisting of representatives of Committees C-9, C-15, and C-18 and set up to provide a means of contact and coordination between the three committees, has not functioned for a number of years.

On the recommendation of Committee E-9 on Research, the Executive Committee has approved the organization of a Research Committee on Impact Properties of Metals. Due principally to the pressure of research activities bearing more directly on production for war, and for other reasons, it has not been possible to proceed with the plans for formal organization, despite the intrinsic importance of this subject.

Other new projects under consideration include a proposal for the Society to organize a new standing committee to deal with standardization in the field of benzene, toluene, zylene, solvent naphthas, light oils, and related materials. A study committee consisting of representatives of the interested standing committees and others is studying the best means of handling such standardization work. A similar study committee is being set up to investigate the desirability of undertaking work in the field of powder metallurgy.

For some time the Executive Commitee has had under consideration the desirability of establishing standard procedures covering magnaflux testing, and has recently included in its consideration methods of testing by means of fluorescent powders. The standing committees interested have been asked for comments and suggestions as a guide to the Executive Committee in reaching a decision in this matter. In the meantime, the Executive Committee has authorized Committee A-1 on Steel to begin work upon the development of standardized procedures for the inspection of heavy forgings by the use of magnetic powders.

Emergency Standardization Procedures:

In connection with the Procedure for Emergency Provisions and Standards given in the Report of the Executive Committee last year,[3] the Executive Committee believes that a more complete report should be presented to Committee E-10 on Standards in connection with any recommendations on emergency alternate provisions or emergency standards; specifically, that the procedure should call for a recorded vote in the subgroup or subcommittee developing the emergency provisions and that reasons for any negative votes should be stated. The following revision has therefore been made in Paragraph 2 of the Procedure, the italicized words indicating additions and those in brackets to be omitted:

> "Such emergency alternate provisions shall first have the approval *by letter ballot* of the appropriate subcommittee of the sponsoring committee or duly appointed subgroup of that subcommittee and shall have the endorsement of the chairman of the main committee. The emergency provisions shall then be submitted to Committee E-10 *together with a covering report including reasons for any negative votes and other comments with respect to the proposal* [for approval for publication]. If approved *by Committee E-10, the emergency alternate provisions* [they] shall be published with the specification either in the form of a sticker or as an accompanying sheet and shall also be published in the next succeeding issue of the ASTM BULLETIN. . . ."

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 56 (1942).

It is pertinent to note that the standing committees have availed themselves of this emergency procedure to a very considerable extent during the past year, so that Committee E-10 on Standards has had a very active year in considering the proposals submitted to it with respect to emergency specifications and emergency alternate provisions.

Marking Requirements in Emergency Alternates:

The Executive Committee has approved the recommendation of Committee E-10 on Standards that a product should be marked with the specific designation of the standard, tentative standard, or emergency alternate to which it conforms, and that in no case should a product be marked as conforming to a standard when in reality it conforms to an emergency alternate whose requirements for quality differ from the standard.

District Committees:

As recorded in the following portions of this report, a number of district committees have sponsored meetings during the year, some of these jointly with other organizations. The new Western New York-Ontario District Committee cooperated with the Buffalo Committee on Arrangements in features of the 1943 Spring Meeting in March. District committees have cooperated throughout the year in various ways including membership promotion and the like. The Pittsburgh District Committee is functioning as a Committee on Arrangements for the 1943 Annual Meeting. The district groups in various centers have on several occasions convened to discuss Society affairs as presented by members of the Headquarters Staff.

Because of the increased importance which district committees are expected to have in the Society, the Executive Committee during the year expanded the scope of one of its Administrative Subcommittees, generally to advise the District officers and to coordinate district committee affairs. In its expanded scope this Executive Subgroup covers District Committees and Promotional Activities.

A new District Committee was organized during the year in the Buffalo-Rochester-Ontario area, as detailed in the January, 1943, ASTM BULLETIN. This brings to ten the number of such district groups. There is very active interest in Society affairs on the part of many individuals in this district and the new committee will foster this interest.

Meetings:

The accompanying table lists various meetings held by the Society and its district committees since the last annual meeting. Several of these meetings were outstanding and a few notes concerning them are desirable for the record.

The technical program, advancement of A.S.T.M. standardization and research work, and attendance at the 1942 Annual Meeting in Atlantic City were notable. The attendance of 1376 registered members, committee members, and guests in a period of emergency such as exists is indicative of the importance accorded the work of the Society.

The 1942 Spring Meeting in Buffalo was a particularly notable one with two very successful technical symposiums: one on paints, in particular civilian defense, blackout, and related protective coatings; the other on powder metallurgy. The several technical papers in each of these symposiums will be compiled in published form in the near future, and members who did not attend the meeting will have an opportunity of noting what an excellent piece of work was done by the Program Committee appointed by the Buffalo Committee on Arrangements. There were upwards of 250 in attendance at the powder metallurgy symposium, with more than 200 at each of the morning and afternoon paint sessions. An interesting dinner program was arranged in the evening by the newly organized Western New York-Ontario District Committee with Captain A. J. Wellings, Head, Navy Material Inspection Administration, as the principal speaker.

A.S.T.M. MEETINGS JUNE, 1942–APRIL, 1943, INCL.

Meeting	Place	Date	Notes
Annual Meeting	Atlantic City, N. J. Chalfonte-Haddon Hall Hotel	June 22–26, 1942	16 Technical Sessions 175 Committee Meetings 1376 Attendance, plus 200 ladies
Spring Meeting	Buffalo, N. Y. Hotel Statler	March 3, 1943	Symposiums on Paints and on Powder Metallurgy Over 200 at each of the three Sessions
A.S.T.M. Committee Week	Buffalo, N. Y. Hotel Statler	March 1–4, 1943	55 Committee Meetings 378 Attendance
District or Local (Sponsored by Groups in Cities Indicated)	Philadelphia	September 24, 1942	Recent Progress in Timber Research by L. J. Markwardt (200 attendance)
	Philadelphia Joint Meeting with American Society for Metals	October 2, 1942	How a German Aircraft Engine Compares with American in Design and Performance by Bishop Clements and R. W. Young (800 attendance)
	Southern California (Los Angeles)	November 13, 1942	General Dinner Meeting and Technical Information (100 attendance)
	Philadelphia Joint Meeting with American Welding Society	November 16, 1942	Stress Distribution by Everett Chapman (150 attendance)
	Pittsburgh	January 11, 1943	Behavior of Glazing Materials Subjected to Explosion by Dr. F. W. Adams Rôle of Specifications in Conservation of Critical Materials by C. L. Warwick (Upwards of 100)
	Philadelphia	January 20, 1943	Round Table Discussion on Significance of Specifications, with following participating: A. O. Schaefer, L. H. Winkler, L. B. Jones, E. L. Hollady, Commander E. C. Forsyth, R. W. Orr (200 attendance)
	Chicago Joint Meeting with Western Society of Engineers (Structural Div.)	February 4, 1943	Recent Progress in Timber Research by L. J. Markwardt (300 attendance)
	Philadelphia	February 10, 1943	Plastics by Captain M. H. Bigelow and Dr. A. Allan Bates (Over 500 attendance)
	Chicago Invitation Meeting with Western Society of Engineers	April 22	Recent Developments in Fuel Supply and Demand by A. C. Fieldner (100 attendance)
	Philadelphia	April 22	Use and Abuse of Gasoline, Kerosine, etc. by Prof. Charles M. Allen (175 attendance)

The outstanding District meeting during the year was undoubtedly the one in Philadelphia on February 10 on the subject of plastics with an overflow attendance of over 500. Other notable meetings were those in Chicago on February 4 and in Philadelphia on September 24, at each of which L. J. Markwardt gave his most interesting talk on timber. The paper by Dr. F. W. Adams on "Behavior of Glazing Materials Subjected to Explosion" presented in Pittsburgh was published in the May BULLETIN. The Philadelphia District Committee continued its very active work in sponsoring several interesting meetings.

In addition to providing an opportunity for members of the Society to meet each other and particularly for new members in their respective districts to have contacts with others in A.S.T.M., the meetings sponsored by district committees have a valuable educational aspect by giving those in attendance a better understanding of some of the essential features of engineering materials problems.

Publications:

This year has surpassed all other years in the history of the Society so far as the magnitude of the publication activities is concerned. This was due primarily to the appearance during 1942 of the triennial Book of Standards and the unprecedented size of this book (total pages, 4906). In addition, practically all of the compilations of standards were published during the past year and two new ones added, namely, Soaps and Detergents, sponsored by Committee D-12, and Plastics, sponsored by Commitee D-20. On this regular publication work was superimposed the printing of 103 Emergency Alternate Provisions in existing standards and 31 complete new Emergency Standards.

As will be noted below the Book of Standards is being continued on the same basis as published in 1939, namely, in three parts: Part I devoted to Metals, Part II Nonmetallic Materials—Constructional and Part III Nonmetallic Materials—General. In each of these both the formally adopted standards and the tentative standards are included. This scheme of publication was worked out for the 1939 edition and proved to be quite a convenience to all users of the Book of Standards and accordingly has been followed in the publication of the 1942 edition.

Despite the increase in size (approximately one third), no change has been made in the cost to members for the copies received by virtue of membership. A slight increase was made in the member's price for extra copies and a proportionately larger increase in the prices to nonmembers somewhat commensurate with the increased size and intrinsic value. These prices were announced currently in the ASTM BULLETIN.

A publication deserving special comment is the 1943 Symposium on Radiography. The Symposium on Radiography and X-ray Diffraction published in 1936 has received recognition as an authoritative publication in the radiographic field. For some months this book was out of print and upon consulting the committee in charge, it was decided that in view of developments in the field of radiography and in the apparatus used, a new Symposium on Radiography should be held. As a result a Symposium on Radiography, consisting of twelve papers by outstanding authorities in the field, was held at the 1942 Annual Meeting. These papers are published in the 1942 *Proceedings*; and the same papers, together with the papers covering the Radiography section of the 1936 Symposium (revised and brought up to date) and a paper by L. S.

Taylor appearing in the ASTM BULLETIN, were published as a separate volume to replace the Radiography section of the 1936 Symposium.

The special Report on Significance of Tests of Concrete and Concrete Aggregates, a revision of the 1935 publication, was completed and copies were available in March, 1943.

As mentioned above, practically all the special compilations of standards were published during the past year. These compilations are listed below:

A.S.T.M. Specifications for Steel Piping Materials (Committee A-1) greatly enlarged in scope, 44 standards, 22 EA stickers, 255 pages, 1600 copies, published annually.

A.S.T.M. Standards on Electrical-Heating and Resistance Alloys (Committee B-4), 23 standards, 145 pages, 800 copies, published annually.

A.S.T.M. Standards on Copper and Copper Alloys (Committee B-5), 87 standards, one ES, 32 EA stickers, 383 pages, 1200 copies, published annually.

A.S.T.M. Standards on Cement (Committee C-1), 13 standards, one ES, 123 pages, 1800 copies, published annually.

A.S.T.M. Standards on Minerals Aggregates, 42 standards, 126 pages, 1000 copies.

A.S.T.M. Standards on Paint, Varnish, Lacquer, and Related Products (Committee D-1) 126 standards, one ES, one EA sticker, 419 pages, 1500 copies, published triennially.

A.S.T.M. Standards on Petroleum Products and Lubricants (Committee D-2), 89 standards, one EA sticker, 452 pages, 5077 copies, published annually.

A.S.T.M. Standards on Coal and Coke (Committee D-5), 27 standards, 127 pages, 1600 copies, published biennially.

A.S.T.M. Standards on Electrical Insulating Materials (Committee D-9), 64 standards, two EA stickers, 452 pages, 900 copies, published annually.

A.S.T.M. Standards on Rubber Products (Committee D-11), 41 standards, 7 EA stickers, 309 pages, 1100 copies, published annually.

A.S.T.M. Standards on Soaps and Other Detergents (Committee D-12), 28 standards, eight EA stickers, 135 pages, 1100 copies.

A.S.T.M. Standards on Textile Materials (Committee D-13), 74 standards, one EA sticker, 421 pages, 1750 copies, published annually.

A.S.T.M. Standards on Plastics (Committee D-20), 70 standards, 383 pages, 1200 copies.

The following regular publications were issued and distributed to the members:

1942 *Proceedings*, 1225 pages, 5100 copies. In addition preprints of reports and papers totaled 1045 pages, 1600 copies.

1942 Book of Standards:

Part I, 1695 pages, plus 6 insert plates, 9500 copies.

Part II, 1528 pages, plus one insert plate, 7400 copies.

Part III, 1681 pages, plus two insert plates, 7400 copies.

Index to Standards (1942–1943), 206 pages, 18,600 copies.

Year Book, 352 pages, 3500 copies.

ASTM BULLETIN, six issues, August, 1942 to May, 1943, 456 pages, average edition, 7100 copies.

A statement of the volume of the principal publications for the past four years follows:

Publications	1939, pages	1940, pages	1941, pages	1942, pages
Proceedings	1352	1403	1388	1225
Book of A.S.T.M. Standards	3794	....	....	4906
Supplements to Book of A.S.T.M. Standards	....	1445	1731	....
A.S.T.M. Methods of Chemical Analysis of Metals	256	....	....	[a]
Index to A.S.T.M. Standards and Tentative Standards	160	172	196	206
Year Book	304	320	336	352
Spring Meeting Symposiums	251	100	202	....
	6117	3440	3853	6689

[a] Nearing completion.

The following special publications were also issued:

The Significance of Tests of Petroleum Products, prepared by Committee D-2, 82 pages (a reprint), 3000 copies.

Tables of Data on Chemical Compositions, Physical and Mechanical Properties of Wrought Corrosion-Resisting and Heat-Resisting Chromium and Chromium-Nickel Steels, sponsored by Committee A-10, 45 pages, 2500 copies.

Report on Significance of Tests of Concrete and Concrete Aggregates, sponsored by Committee C-9, 177 pages, 2500 copies, second edition.

Round-Table Discussion on the Solvent Action of Water Vapor at High Temperature and Pressure (a reprint from *Proceedings*), 45 pages, 3500 copies.

Symposium on Radiography, 262 pages, 3000 copies.

A.S.T.M. Manual on Presentation of Data, 83 pages (a reprint), 3000 copies.

The Book of A.S.T.M. Methods of Chemical Analysis of Metals, last published in September, 1939, is now being completely restyled, the standards brought up to date, and a number of new methods added. Work on this book is nearing completion.

The Symposium on Mineral Aggregates mentioned in last year's report has been deferred, due to the war effort, until a propitious time for the completion of the papers to be included in it.

responsible for censorship. Export licenses have been granted covering practically all of the Society's publications, although in a number of instances distribution is restricted to certain countries, which explains why certain of our members in foreign countries have not yet received some of the Society's publications.

Membership:

The membership of the Society on June 1, 1943, was 4904, which is the highest in its history. Statistics showing gains and losses in various classes of membership are shown in the following table:

Class of Membership	Membership		Losses				Additions		Total		
	June 1, 1942	June 1, 1943	Resignation	Dropped	Death	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	7	7	...	...	..	..	..	...	...	...	..
Perpetuity	9	9	...	...	..	..	..	...	...	...	..
Sustaining	148	165	...	...	..	1	12	6	1	18	17
Company, Firm, etc.	1070	1162	19	13	..	13	5	132	45	137	92
Individual, etc.	3306	3444	90	66	30	4	20	308	190	328	138
Junior	100	117	6	2	..	19	..	44	27	44	17
Total	4640	4904	115	81	30	37	37	490	263	527	264
Student	419	413	27	266	..	24	..	311	317	311	6[a]

[a] Loss.

An index of the compounds covered in the X-ray Diffraction Data Cards has been published for free distribution to all purchasers of the sets of data cards.

It was also found necessary to reproduce an additional 100 sets (20 photographs each) of the Cotton Yarn Appearance Standards. These standards were made available by the Society starting last year.

In common with all other products, the export of the Society's publications is subject to approval on the part of the Government. Each of these publications upon its appearance is submitted to the Board of Economic Warfare which is

The net gain for the year of 264 compares with 170 for the preceding year. Elections to membership were close to the highest in the history of the Society and numbered 490, exceeding those for the previous year by 116; losses from resignations and delinquencies were 196 compared to 183 for the preceding year. The number of Junior Members was 117 and of student members was 413.

There has been a net increase of 17, in the number of sustaining members bringing the total to 165. There were 18 new sustaining members and one loss. Of the 18, twelve were acquired by transfer of existing company memberships and six are companies which had not previously held membership in the Society, except the National Lead Co. which has for some years held a membership in perpetuity and has now also acquired a sustaining membership. The names of the new sustaining members are as follows:

Baker and Co., Inc.
Calumet Refining Co.
Crane Co.
Gulf Research and Development Co.
Johns-Manville Corp.
Lever Brothers Co.
Liquid Cooled Engine Division, The Aviation Corp.
Lone Star Cement Corp.
Lunkenheimer Co., The
Matam Corp.
Meyer Engineering Co.
Mica Insulator Co.
National Lead Co.
National Malleable and Steel Castings Co.
Peoples Gas Light and Coke Co.
Phelps Dodge Refining Corp.
United Aircraft Corp.
Western Union Telegraph Co., The

The Executive Committee expresses on behalf of the Society its thanks to these companies for their willingness to lend increased financial support to the Society's activities through this form of membership.

The Executive Committee also expresses its thanks to the district committees and the many members of the Society who throughout the year have aided so effectively in the acquiring of new members.

As announced two years ago, the Executive Committee has authorized the Secretary-Treasurer to waive upon their request the dues of members of the Society who have entered the Armed Forces of their country. The names of such members are retained on the membership rolls during the period of their service and they will retain all privileges of membership, save that the Book of Standards and the *Proceedings* will not be sent them. They will continue to receive the ASTM BULLETIN. Only 21 members of those now in the Armed Services have requested such waiver of dues. Regarding membership on standing committees of members in service, the standing committees are privileged to take cognizance of any unusual conditions of this kind affecting the activity of their committee members and, if they desire, to place such members in an inactive status for the duration of the emergency.

The Society has lost the following members through death:

	Date of Membership
Adams, Walter S.	1920
Badger, A. C.	1914
Danforth, Raymond Hewes	1916
Ellis, Arthur E.	1919
Finn, Alfred N.	1937
Foster, Benjamin	1929
Franklin, Frederick H.	1937
Gardner, Edmund S.	1937
Greenman, Russell S	1904
Greiner, J. E.	1902
Haering, David W.	1941
Hartman, Ernest F.	1914
Hertz, Stanton	1933
Johnson, John A.	1942
Kaltenbach, Robert W.	1929
LaPorte, C. W.	1927
Leschen, Harry J.	1926
Linton, Robert	1915
Lunn, Edward G.	1938
Newlin, John A.	1913
Prentice, E. B.	1924
Smith, E. W. P.	1936
Smith, Noah B.	1918
Swanger, William H.	1937
Teller, George L.	1926
Upton, George Burr	1914
Vaughan, H. H.	1915
Vidal, Emile N.	1941
Wooldridge, William J.	1923
Worcester, Joseph R.	1904

The list of deceased members includes many who were active in the committee work of the Society, among them Russell S. Greenman formerly Chairman of Committee C-1 on Cement and John

A. Newlin, Secretary of Committee D-7 on Timber.

In 1938 the Executive Committee established the plan of awarding at the annual meetings certificates commemorating forty years of continuous membership in the Society. Forty three such certificates have been awarded and this year eighteen such awards will be made to the following members:

Frederick W. Bateman
The Carpenter Steel Co.
Almon H. Fuller
Harvey G. Kittredge
Arthur D. Little, Inc.
J. Strother Miller
Rudolph Philip Miller
Leon S. Moisseiff
Herbert F. Moore
National Lead Co.
Pennsylvania State Highway Dept.
Henry H. Quimby
Charles Edward Skinner
Maximilian Toch
Enrique Touceda
C. P. Van Gundy
Hermann von Schrenk
L. W. Walter

Finances:

Report for the Fiscal Year 1942.—The annual statement of the finances of the Society follows in the report of the auditors for the fiscal year, January 1 to December 31, 1942. The report gives the balance sheet of assets and liabilities, including special and designated funds (that is, the Research Fund, Medal and Lecture Fund, and Committee Funds); statement of receipts and disbursements, in which items are classified into "operating" (budgeted) and "nonoperating"; details of special funds; and investment of Society funds, both book and market values being given.

Total receipts from dues and entrance fees in 1942 were $101,461.23, an increase of $4600 over the preceding year. Receipts from sales of publications totaled $88,181.86 and reflect the continued and increasing demand for the Society's publications. The outstanding items are the sales of the Book of Standards and its Supplements, $30,001.37; the special compilations of standards in specific fields, $13,822.95; the sale of X-ray Diffraction Data Cards for Chemical Analysis, $6228.06. Miscellaneous receipts amounted to $18,100.94, consisting principally of receipts from advertising in the ASTM BULLETIN and Index to Standards, receipts from interest and dividends, and registration fees.

Total operating receipts were $207,744.03. Total operating disbursements with all current bills paid (as detailed in the auditors' report) were $204,043.05, which includes $1800 (one half of the entrance fees) paid into the principal of the Research Fund, $1643.10 paid towards the Employees' Retirement Plan and $572.58 war emergency expense. Thus a favorable balance between operating receipts and disbursements of $3700.98 has resulted.

The auditors' report includes a statement of the financial condition of the Society for the past five years. The surplus in the general funds account, exclusive of special and designated funds, is $84,577.11. During the year $1500 was added to the Headquarters Fund, bringing the total to $6676.48. The sum of $2000, earmarked for future publication of X-ray diffraction data cards, was added to the Publication Fund, bringing the total to $5015. Also there was added $1375 to the Executive Retirement Reserve, totalling $3120. The reserve for the Book of Standards at the close of the year was $13,890.25; when payment for the 1942 Book of Standards and the emergency alternate provisions and specifications was completed in 1943 the sum of $11,776.27 was used from the reserve, leaving a balance of $2113.98 which has been placed in the reserve for the 1945 edition.

During the year $1600 was transferred from surplus to the principal of the Research Fund; to this was added $1800 (one half of the current entrance fees), bringing the total of principal in this fund to $33,548.69.

The cost of the three parts of the 1942 Book of Standards was $41,789.46. The emergency alternate specifications and provisions that form a part of this publication cost an additional $2056.06, bringing the total to $43,845.52. Of this amount $32,069.25 was paid from 1942 current funds; the balance of $11,776.27 was paid from reserve funds as noted above.

Investment of Society funds as of December 31, 1942, are given in the auditors' report, both book and market values being shown. The following changes during the fiscal year in the investment portfolio are noted:

GENERAL FUNDS	
Sold	*Received*
$2000 North Station Industrial Building Bonds, 5s, 1962	$1723.02
Bought	*Cost*
20 shares G. C. Murphy Co. 4¾ pfd.	$2100.00
3 shares American Cyanamid Co. 5% Cumulative Preference (dividend)	....
$5000 U. S Defense Savings Bonds Series G	$5000.00
RESEARCH FUND	
Sold	*Received*
$3000 North Station Industrial Building Bonds, 5s, 1962	$2570.04
Bought	*Cost*
30 shares G. C. Murphy Co. 4¾ pfd.	$3150.00
$3000 U. S. Defense Savings Bonds Series G	3000.00

No account has been taken in the accompanying financial statement of the assets of the Society in the form of publications in stock. An inventory of technical publications as of June 1, 1943, may be summarized as follows:

		Number of Copies
Year Book		125
Proceedings		5 181
Index to Proceedings, 6 parts, total		2 146
1942 Book of A.S.T.M. Standards:		
Part I	2897	
Part II	2072	
Part III	2191	
	——	7 160
Index to A.S.T.M. Standards		4 760
Selected Standards for Students in Engineering		2 772
Reprints of Standards (approximate)		85 000
Special Reprints (approximate)		42 000

1943 Finances.—The Society's operations for 1943 are based upon budgeted operating receipts of $215,000. Receipts from dues and entrance fees are estimated at $101,000; receipts from sales of publications at $99,500, of which about $47,000 is expected to be realized from sales of the Book of Standards and its Supplements; miscellaneous receipts, including advertising, interest and dividends are estimated at $14,500. Disbursements have been estimated at $213,800, leaving $1200 for contingencies. The budget has been prepared on a more conservative basis than usual, in view of the abnormal conditions existing. It provides fully for the normal operating expenses of the Society, including current publications, emergency specifications, activities of standing and district committees, increased rent for the expanded headquarters, as well as required additions to the clerical staff. The budget includes a reserve of $8000 to be applied toward the cost of publishing the 1945 Book of Standards and $1000 toward expenses that may be incurred in connection with the war emergency program.

The Executive Committee reviews the budget of receipts and disbursements quarterly and makes any adjustments that may be necessary. From the surplus at the end of 1942, the Executive Committee has transferred $3000 to the Executive Retirement Reserve and $3000 to the Retirement Fund Reserve, as noted in the following section of this report. Also, a reserve of $10,000 has been established from surplus against depreciation of investments in special and designated funds, namely, Research Fund, Medal and Lecture Fund, and Committee Funds.

Since January 1, 1943, securities have been sold yielding $12,614.93 and investments have been made in the sum of

REPORT OF THE AUDITORS FOR THE FISCAL YEAR, JANUARY 1, TO DECEMBER 1, 1942

Philadelphia, January 11, 1943.

MR. C. L. WARWICK, *Secretary-Treasurer*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pennsylvania

Dear Sir:

We have examined the books and accounts of your Society for the period from January 1, 1942, to December 31, 1942. We did not make a detailed audit of all transactions but made extensive tests to determine the accuracy of your records.

We have prepared and submit herewith Balance Sheet as of December 31, 1942, Statement of Cash Receipts and Disbursements for the year ended that date, and other supporting schedules, all of which are in agreement with the records of your Society. In our opinion, these statements present, fairly, the financial position of the Society at December 31, 1942, and the results of its operations for the year then ended.

The investments owned by the Society, as detailed herein, were examined by us. All income from investments was properly accounted for.

Respectfully submitted,

JOHN HEINS & CO.
(Signed) I. RUSSELL BUSH
Certified Public Accountant

BALANCE SHEET AS OF DECEMBER 31, 1942
(Including Special and Designated Funds)
ASSETS

General Funds:			
Current: Cash	$58 915.67		
Less checks drawn as of December 31, 1942—Cost of Proceedings ($14 500.00) and 1942 Book of Standards, and Emergency Alternate Provisions ($29 500.00) in hands of Secretary-Treasurer that date	44 000.00		
		$14 915.67	
Investments (Market Value, $76 508.76)—Cost		93 281.45	
Accounts Receivable		6 846.09	
Total Current Assets—General Fund			$115 043.21
Furniture and Fixtures (depreciated book value)			6 362.86
Total General Funds			$121 406.07
Special and Designated Funds:			
Cash: A.S.T.M. Research Fund		$1 642.32	
Dudley Medal and Marburg Lecture Fund		694.93	
Committee Funds		7 161.30	9 498.55
Investments:			
A.S.T.M. Research Fund (Market Value, $27 024.88)—Cost or book value		$32 693.49	
Dudley Medal and Marburg Lecture Fund (Market Value, $2 136.25)—Cost		6 625.00	
Committee Funds (Market Value, $6 331.57)—Cost		6 000.00	
			45 318.49
Total Special and Designated Funds			$54 817.04
Total Assets			$176 223.11

LIABILITIES

General Funds:			
Current: Accounts Payable:			
Committee A-1	$11.33		
Committee C-1	174.85		
Advance Collections	4 850.10		
Total Current Liabilities—General Fund		$5 036.28	
Life Membership Fund	$3 005.20		
Headquarters Fund	6 676.48		
Publication Fund	5 015.00		
Book of Standards Reserve	13 890.25		
Executive Retirement Reserve	3 120.00		
Retirement Fund Reserve	85.75		
Surplus	84 577.11		
		116 369.79	
Total General Funds			$121 406.07
Special and Designated Funds:			
A.S.T.M. Research Fund:			
Principal	$33 548.69		
Income	787.12	$34 335.81	
Dudley Medal and Marburg Lecture Fund:			
Principal	6 625.00		
Income	694.93	7 319.93	
Committee Funds		13 161.30	
Total Special and Designated Funds			54 817.04
Total Liabilities and Funds			$176 223.11

COMPARISON OF GENERAL FUNDS FOR FISCAL YEARS 1938–1942, INCLUSIVE

At Close of Fiscal Year	Assets				Liabilities			
	Cash	Investments	Accounts Receivable and Miscellaneous	Furniture and Fixtures	Accounts Payable and Miscellaneous	Reserve for Book of Standards	Miscellaneous Funds	Surplus
1938	$24 425.51	$39 568.24	$9 609.00	$6 912.96	$2 283.88	$12 000.00	$8 898.99	$57 332.84
1939	9 329.65	54 902.10	14 880.53	6 467.27	5 777.01	12 000.00	9 685.40	58 117.14
1940	9 608.61	72 568.95	13 388.88	6 561.93	3 380.25	7 890.25	9 713.79	81 144.08
1941	18 206.61	88 001.45	14 120.99	6 440.58	3 937.44	13 890.25	12 907.43	96 034.51
1942	14 915.67	93 281.45	6 846.09	6 362.86	5 036.28	13 890.25	17 902.43	84 577.11

RECEIPTS AND DISBURSEMENTS
FOR THE PERIOD ENDED DECEMBER 31, 1942

Cash balance, January 1, 1942			$18 206.61
RECEIPTS			
Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$94 431.30		
Past Dues	585.50		
Advance Dues	2 704.43		
Income, Life Membership Fund	150.00		
Entrance Fees	3 590.00		
Total Dues		$101 461.23	
Sale of Publications:			
Separate Standards	8 771.20		
Book of Standards (Members, additional parts)	6 075.00		
1939 Book of Standards	14 078.12		
1940 Supplement to Book of Standards	4 406.98		
1941 Supplement to Book of Standards	10 523.67		
1942 Book of Standards	992.60		
Methods of Chemical Analysis of Metals	1 024.11		
Proceedings	3 229.05		
Index to Papers and Reports	461.60		
Selected Standards for Students in Engineering	4 235.25		
Bulletin Susbcriptions (Nonmembers)	427.85		
Special Reprints and Miscellaneous	33 956.43		
Total Sale of Publications		$88 181.86	
Miscellaneous:			
Binding (Members)	191.60		
Advertising, Bulletin	8 564.87		
Advertising, Index	1 910.52		
Interest and Dividends	4 688.36		
Registration and Other Fees, Annual Meeting	1 397.00		
Committee A-1 for Technical Assistant	1 009.28		
Miscellaneous	339.31		
Total Miscellaneous Items		$18 100.94	
Total Operating Receipts (Budgeted)		$207 744.03	
Nonoperating Receipts (Not Budgeted):			
Investments, Matured or Sold	$1 723.02		
Excess Remittances	260.83		
Committee C-1 for Technical Assistant	1 500.00		
Executive Retirement Reserve Income	120.00		
Total Nonoperating Receipts		$3 603.85	
Total Receipts			$211 347.88
Total Receipts and Cash Balance January 1, 1942			$229 554.49
Brought forward			$229 554.49

DISBURSEMENTS

Operating Disbursements (Budgeted):			
Publications:			
Year Book	$2 498.93		
1939 Book of Standards	2 010.73		
1941 Supplement to Book of Standards	379.78		
1942 Book of Standards	29 069.25[a]		
Methods of Chemical Analysis of Metals	10.98		
Proceedings	16 967.75[b]		
Preprints	3 136.61		
Spring Meeting Papers	399.99		
ASTM Bulletin	9 531.54		
Circulars to Members	1 395.17		
Separate Standards	4 090.74		
Emergency Alternate Provisions	5 891.28[a]		
Selected Standards for Students	1 382.44		
Index to Standards and Tentative Standards	3 353.39		
Collective Index to Papers and Reports	1 236.70		
Reprints (Reports, Papers, Symposiums, etc.)	12 890.99		
Miscellaneous Printing (Authors' Reprints, etc.)	939.43		
Total Disbursements, Publications		$95 185.70	
Salaries		72 246.67	
General Office Expenses		14 371.29	
Expenses, Standing and District Committees		3 504.78	
Expenses, Meetings		3 028.13	
Traveling Expenses, Administrative and Special Committees		3 153.95	
American Standards Assn. and Sectional Committees		1 000.00	
Rent, Headquarters		6 250.00	
Furniture and Fixtures		886.50	
Principal, A.S.T.M. Research Fund (one half current year's entrance fees)		1 800.00	
Employees Retirement Fund		1 643.10	
Emergency National Defense Expense		572.58	
Miscellaneous (Dues, Contributions, etc.)		400.35	
Total Operating Disbursements (Budgeted)		$204 043.05	
Nonoperating Disbursements (Not Budgeted):			
Investments	$7 100.00		
Refund of Excess Remittances	260.83		
Technical Assistant, Committee C-1	1 634.94		
Principal, A.S.T.M. Research Fund	1 600.00		
Total Nonoperating Disbursements		$10 595.77	
Total Disbursements			$214 638.82
Cash Balance, December 31, 1942			$14 915.67

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[a] This amount includes $29 500.00 covering checks drawn against cost of 1942 Book of Standards, and Emergency Alternate Provisions, but not actually paid on December 31, 1942.

[b] This amount includes $14 500.00 covering checks drawn against cost of *Proceedings*, but not actually paid on December 31, 1942.

In addition to the general funds of the Society, the Secretary-Treasurer has on hand the following funds as of December 31, 1942:

A.S.T.M. RESEARCH FUND

	Total	*Invested*	*Uninvested Cash*
Principal Account:			
Balance January 1, 1942	$30 391.15	$29 355.99	$1 035.16
Receipts:			
Contribution—A.S.T.M. Current Entrance Fees	+1 800.00		+1 800.00
Contribution—A.S.T.M. Past Entrance Fees	+1 600.00	−2 570.04	+1 600.00
Loss on Sale of Securities	−242.46	−242.46	+2 570.04
	$33 548.69	$26 543.49	$7 005.20
Disbursements: Purchase of Securities		+6 150.00	−6 150.00
Balance, December 31, 1942	$33 548.69	$32 693.49	$855.20
Income Account:			
Balance January 1, 1942	$1 256.95		
Receipts:			
Interest and Dividends	1 080.17		$2 337.12
Disbursements:			
Research Committee on Fatigue of Metals	$1 000.00		
Joint Research Project on Durability of Concrete (Highway Research Board)	250.00		
Welding Research Committee for the Carbon Manganese (Weldability) Program (Engineering Foundation)	300.00		$1 550.00
Cash Balance, December 31, 1942			$787.12

DUDLEY MEDAL AND MARBURG LECTURE FUND

Balance, January 1, 1942:			
Principal—Investments (at cost)	$6 625.00		
Income—Cash	596.80	$7 221.80	

Receipts

Interest on Investments	378.38	$7 600.18

Disbursements

Engrossing 1942 Lecture Certificate	$3.25	
Honorarium, 1942 Lecturer	200.00	
Cost of 1942 Medals	77.00	280.25
		$7 319.93
Balance, December 31, 1942:		
Principal—Investments (at cost)	$6 625.00	
Income—Cash	694.93	$7 319.93

COMMITTEE FUNDS

Committee A-1 on Steel	$671.09	
Committee A-5 on Corrosion of Iron and Steel	3 065.83	
Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys	2 981.56	
Committee B-6 on Die-Cast Metals and Alloys	84.30	
Committee C-1 on Cement	813.26	
Committee C-1—Cement Reference Laboratory	961.29	
Committee C-9 on Concrete and Concrete Aggregates	195.12	
Committee C-9—Sanford E. Thompson Medal Fund	429.45	
Committee C-15 on Manufactured Masonry Units	341.36	
Committee C-18 on Natural Building Stones and Slate	172.36	
Committee D-1 on Paint, Varnish, Lacquer, and Related Products	82.00	
Committee D-2 on Petroleum Products and Lubricants	122.37	
Committee D-5 on Coal and Coke	1 261.10	
Committee D-13 on Textile Materials	509.52	
Committee D-13—Testing Textile Finishes	268.27	
A.S.A. Sectional Committee on Specifications for Cast-Iron Pipe and Special Castings (Overdraft)	43.83	
Research Committee on Fatigue of Metals	79.05	
Detroit District Committee	1 167.20	
Total Committee Funds		$13 161.30
Accounted for as follows:		
Cash Balance, December 31, 1942	$7 161.30	
Investments—at cost	6 000.00	$13 161.30

AMERICAN SOCIETY FOR TESTING MATERIALS INVESTMENTS (AS OF DECEMBER 31, 1942)

Number of Shares or Par Value		*Cost or Book Value*	*Market Value*
	GENERAL FUNDS		
	Bonds		
$3000	Armour & Co., of Del., First Mortgage, 20-yr., Sinking Fund, Series C, 4's, due 1957	$2 955.00	$3 097.50
9000	Balto. & Ohio R. R., Refunding and General Mortgage 5's, Series F, due 1996	9 106.55	2 936.25
2000	Columbia Gas & Electric Co., Debentures, 5's, due 1961	2 025.34	1 745.00
500	Florida Power & Light Co., First Mortgage, 5's, due 1954	491.25	521.25
4000	General Steel Castings Corp., First Mortgage, Series A, 5½'s, due 1949	3 610.00	3 970.00
3000	Michigan Consolidated Gas Co., First Mortgage, 4's, due 1963	2 925.00	3 202.50
4000	N. Y., Chicago & St. Louis R. R., Refunding Mortgage, Series C, 4½'s, due 1978	2 805.61	2 580.00
3000	Pressed Steel Car Co., Inc., 15-yr. Debentures, 5's, due 1951	2 868.75	2 876.25
7000	U. S. Defense Savings Bond, Series G, due July 1, 1953[b]	7 000.00	7 000.00
3800	U. S. Treasury Bonds, 2½'s, due 1967–72	3 800.00	3 821.38
		$37 587.50	$31 750.13
	Stock		
30	Alvin Progressive Federal Savings & Loan Assn.[b]	$3 000.00	$3 000.00
25	American Can Co., common	2 122.30	1 793.75
16	American Cyanamid Co., 5 per cent Cumulative preference	160.00	184.00
50	American Cyanamid Co., Class B common	1 764.25	1 993.75
100	American Radiator & Std. Sanitary Corp., common	1 564.32	600.00
25	American Tobacco Co., common	2 110.12	1 056.25
100	Atlantic Refining Co., common, par value $25	2 302.50	1 875.00
100	Borden Co.	1 989.00	2 275.00
25	Colgate-Palmolive-Peet Co., $4.25 preferred	2 525.00	2 675.00
30	Collingdale Federal Savings & Loan Assn.[b]	3 000.00	3 000.00
50	Gen. Elec. Co. common, no par value	2 006.25	1 525.00
50	General Telephone Corp., $2.50 preferred[a]	2 675.00	2 200.00
50	Great Northern Ry., $6 preferred par value $100	1 774.83	1 125.00
25	Hazel Atlas Glass Co.	2 510.64	2 362.50
100	S. S. Kresge Co., common	2 490.18	1 875.00
100	Mathieson Alkali Works, common, no par value	3 016.00	2 100.00
20	G. C. Murphy Co., 4¾ per cent preferred	2 100.00	2 260.00
25	J. C. Penney Co., common	2 279.63	2 025.00
100	Public Service Corp. of N. J., common	4 129.50	1 162.50
25	Sears, Roebuck & Co., Capital	1 760.71	1 546.88
200	United Gas Improvement Co., common	2 578.00	1 150.00
50	U. S. Gypsum	3 819.27	3 050.00
40	Westinghouse Elec. & Mfg. Co., common	3 456.45	3 270.00
8	Westinghouse Elec. & Mfg. Co. (dividend shares)	560.00	654.00
		$55 693.95	$44 758.63
		$93 281.45	$76 508.76

	A.S.T.M. RESEARCH FUND		
	Bonds		
$2000	First Mortgage Real Estate, 1315–1317 Spruce St., 6's, due 1945[a]	$2 000.00	$700.00
1000	Allis-Chalmers Mfg. Co., Convertible Debentures, 4's, due 1952	1 035.28	1 072.50
1500	Balto. & Ohio R. R., General & Refunding, 5's, Series F, due 1996	1 511.25	489.38
2000	Columbia Gas & Electric Co., Debentures, 5's, due 1961	2 025.31	1 745.00
1500	Florida Power & Light Co., First Mortgage, 5's, due 1954	1 473.75	1 563.75
2000	N. Y., Chicago & St. Louis, Refunding & Mortgage, Series C, 4½'s, due 1978	1 400.26	1 290.00
3000	Republic Steel Corp., General Mortgage, Series B, 4½'s, due 1961	3 000.00	3 000.00
		$15 445.85	$12 894.38
	Stock		
12	Consolidated Edison Co. of N. Y. $5 preferred	$1 234.39	$1 092.00
29	Consumers Power Co. $4.50 preferred, no par value	2 914.50	2 581.00
98	General Motors Corp., common, par $10[c]	6 504.75	4 348.75
30	G. C. Murphy Co., 4¾ per cent preferred	3 150.00	3 390.00
25	Pittsburgh Plate Glass Co., common	1 955.00	2 143.75
100	United Gas Improvement Co., common	1 489.00	575.00
		$17 247.64	$14 130.50
		32 693.49	27 024.88
	DUDLEY MEDAL & MARBURG LECTURE FUND		
$500	Balto. & Ohio R. R., Refunding & General Mortgage 6's, Series C, due 1995	$550.00	$178.75
6000	Balto. & Ohio R. R., Refunding & General Mortgage 5's, Series A, due 1995	6 075.00	1 957.50
		$6 625.00	$2 136.25
	COMMITTEE FUNDS		
$5000	U. S. Treasury Bonds, 2¾'s, due 1947–45	$5 000.00	$5 240.63
1000	U. S. Treasury Bonds, 2⅞'s, due 1960—55	1 000.00	1 090.94
		$6 000.00	$6 331.57
	Grand Total	$138 599.94	$112 001.46

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[a] Market values were taken from current financial publications with the exception of quotations on inactive or unlisted securities indicated by footnote.[a] These values were obtained from brokers as of December 31, 1941.

[b] In the absence of a quoted market value, these securities are included at their cost which is equal to their face value.

[c] The income from these shares is, by agreement, paid to an annuitant.

$12,520, including $9500 invested in Government bonds. The total book value of Society investments as of May 31, 1943, in all funds was $138,907.82. The market value of these securities on the same date was $125,416.64. The Society's cash balance on May 31, 1943, was $115,979.57.

Retirement Plan for Employees:

The Retirement Plan for A.S.T.M. Employees has now been in effect for five years and includes 18 of the 22 employees eligible for participation. With the exception of several of the newer members of the staff, all participants are contributing from 2½ to 3 per cent of their salaries; the Society's contribution for 1942 of $1643.10 is slightly under 2½ per cent of the salary roll.

At its meeting in April the Executive Committee after careful consideration decided that a point has been reached in the history of the Retirement Plan which now justifies substantial extension through recognition of long years of service, thus implementing the principle of recognition of "long time service" inherent in the basic retirement plan. Accordingly retirement policies were taken out for three employees who have over 20 years of service, with annual premiums totaling $525. With this addition and after certain adjustments due to cancelled policies and an initial one for a new employee, the Society's contribution is at the rate of $2150 a year, which is approximately 2.8 per cent of the present salary roll.

The Executive Committee has also added the sum of $3000 to the Retirement Fund Reserve for the three-fold purpose of (1) providing funds that can be used if necessary to support Retirement Plan payments at a time of financial stress; (2) providing for future expansion of the Plan; and (3) anticipating the possibility that the Social Security Laws may be so amended as to include the Society and its staff within their scope.

Expansion of A.S.T.M. Headquarters:

Beginning January 1, 1943, the Society leased on a year-to-year basis two additional rooms adjoining its present offices, bringing the office space to 3865 sq. ft. and storage and shipping space to about 1165, a total of 5030 sq. ft. This figure is about 70 per cent greater than the office and storage space first leased in the Atlantic Building when the Society in 1933 moved its offices from the Engineers' Club of Philadelphia, where they had been for many years.

The space recently taken is being used for the Standards Department of the staff, thus relieving serious congestion elsewhere in the offices; for a retiring room for women employees; and for additional storage space for publications.

Cooperative Activities:

The Society's cooperative relations with other organizations have continued along well-established lines, which for the most part take the form of participation in the work of the Society's technical committees. Such cooperative contacts have been maintained from the inception of the Society's work. An outstanding example of this cooperation is the establishment of a new Joint Committee on Filler Metal with the American Welding Society and the A.S.T.M. as joint sponsors. The authorization of this committee was referred to in the Report of the Executive Committee last year and organization has since been effected.

In order that cooperative relations with the Society of Plastics Industry might be placed on a firmer foundation, meetings were held between representatives of the Society's Executive Committee and the Board of Direction of the S.P.I. As a result it was agreed that the A.S.T.M. would be responsible for the development of testing methods and the writing of specifications, whereas the S.P.I. would devote its attention to the preparation and distribution of engineering data on molded parts, the standardization of dimensions and tolerances, the choice of materials for particular applications, and the standardization of commercial molding techniques. Both organizations, as well as the Plastic Materials Manufacturers Association, are interested in the assembling and dissemination of data on the properties of plastics. Provision has been made for a coordinating committee consisting of representatives of the two organizations to which questions concerning jurisdiction may be referred.

The Executive Committee is pleased to be able to report that these cooperative relations have been established in such an important field as plastics.

Many plastics are of interest to the automotive industry. Cooperative relations have accordingly been established with the Society of Automotive Engineers and this organization has designated representatives to serve with A.S.T.M. Committee D-20 on Plastics. These representatives have been particularly active in the work undertaken recently on nonrigid plastics which have important applications in automotive work.

The Society's contacts with the American Standards Association continues to constitute one of its most important cooperative activities. The principal work has been more or less routine in nature, involving the submission of new and revised standards to the ASA for approval as American Standards, which is presented in detail in the annual report of Committee E-10 on Standards.

The American Standards Association has entered into a contract with the Office of Emergency Management to develop standards that are urgently needed in certain fields, these standards being developed under its war standards procedure. One of the chief activities being carried out under this arrangement is the development of standards in the field of radio and radar, which are of interest to the armed services, particularly the communications branches. The work is under the sponsorship of the War Production Board. So far as materials are concerned, the A.S.T.M. committees that have developed standards that may be of value in this work are primarily Committees D-9 on Electrical Insulating Materials and D-20 on Plastics. In order that the work on war radio may be tied in effectively with the A.S.T.M. committees, a number of individuals have been designated to serve in a liaison capacity—L. J. Cavanaugh so far as electrical insulating materials in general are concerned and M. P. Davis, W. A. Zinzow, Robert Burns, L. W. A. Meyer, and G. M. Kline with respect to work on plastics. Other members of the Society are serving on various materials subcommittees in the radio and radar program. This work, too, has resulted in the preparation of several A.S.T.M. specifications for plastics at the request of the Signal Corps, U. S. A.

In addition to the work on radio and radar, other standards have been requested on safety clothing, this work being carried out under the Office of Price Administration. Here again there may be certain questions on materials concerning which the Society can contribute and accordingly the work is being followed very closely.

In all of this work, it is recognized that the A.S.T.M. may have much to offer or be able to contribute directly, and accordingly there is a general understanding that with respect to any materials questions the A.S.T.M. should be consulted.

H. H. Morgan has been reappointed for a term of three years as the Society's representative on the ASA Standards Council; and M. P. Davis and A. G. Ashcroft have been appointed, respectively, to ASA Sectional Committee C-59 on Electrical Insulating Materials and the ASA Advisory Committee on Ultimate Consumer Goods.

Reference was made in the report a year ago to certain discussions that were then taking place in the American Standards Association with reference to contacts with other American Republics. Since then financial aid has been secured which has permitted the American Standards Association to place this activity on a workable basis. A South American representative has been engaged who is in a position to visit various South American countries and to discuss with them various standardization problems. An engineer and other office assistants are now located at the ASA Headquarters to handle these various inter-American contacts. In addition to assisting standardization committees in the several countries, arrangements have been established for forwarding to this country in draft form standardization proposals that are under consideration. These are brought to the attention of interested groups in this country. The Society is taking part in this work.

Respectfully submitted on behalf of the Executive Committee,

H. J. BALL,
President.

C. L. WARWICK,
Secretary-Treasurer.

JUNE, 1943.

REPORT OF COMMITTEE A-1

ON

STEEL

Although for the first time in many years, Committee A-1 on Steel is not recommending in its report a large number of actions for Society approval, nevertheless it has had a very active year. A large number of emergency alternate provisions and several emergency specifications have been approved as discussed below. One new tentative specification covering Heat-Treated Carbon-Steel Bolting Material (A 261 – 43 T) was also approved.

One reason that there are not more actions on standards being referred to the Society this year is the very intensive activity during 1941–1942 when the committee reviewed all its specifications prior to their publication in the 1942 Book of A.S.T.M. Standards. The committee has been cognizant of the desirability of "freezing" specification requirements, thus keeping changes during the emergency to a minimum; and by formal action at its January, 1943, meeting, the committee went on record as recommending that changes and modifications in existing specifications should not be developed unless they were extremely essential and important in advancing the war effort.

Meetings.—Two series of meetings have been held during the year: the first in Atlantic City, N. J., on June 22 and 23, 1942, and the second in Philadelphia, Pa., on January 20 and 21, 1943. The committee did not participate in A.S.T.M. Committee Week in March, having concentrated its work at the January meeting, with the further expectation that there will be numerous meetings at the June, 1943, Society meeting in Pittsburgh. All meetings have been exceptionally well attended.

Personnel.—Since the 1942 annual meeting, 12 new members have been elected and 2 resignations accepted, the committee membership now totaling 242, of whom 129 are classified as nonproducers and 113 as producers; of the former, 94 are consumers and 35 are general interest members.

Losses by Death.—During the year, Committee A-1 has lost by death the following members: A. C. Badger, Youngstown Sheet and Tube Co.; A. D. Sanderson, Standard Oil Development Co.; E. W. P. Smith, Lincoln Electric Co.; W. H. Swanger, National Bureau of Standards; and John F. Wyzalek, Hyatt Bearing Division, General Motors Corp. Each of these men has contributed in advancing various phases of the committee's work, and the committee records its appreciation of their active interest and support. Mr. Badger was particularly active in the work of the subcommittee concerned with pipe and tubing, and Mr. Sanderson had been very actively interested for many years in the specifications in the charge of Subcommittee IX on Steel Tubing and Pipe and Subcommittee XXII on Valves, Fittings, Piping and Flanges for High-Temperature Service.

RECOMMENDATIONS AFFECTING STANDARDS

Committee A-1 is submitting revisions in three standards and one tentative standard, and is recommending the adoption as standard without revision of two tentative standards as listed in Table I. In addition editorial changes in six standards and tentative standards are being presented. The revisions and editorial changes being recommended are set forth in the Appendix.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVE STANDARD			
Spec. for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 – 42 T)	88	0	69
II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Carbon-Steel and Alloy-Steel Ring and Disk Forgings (A 243 – 41 T)	83	1	60
Spec. for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 41 T)	99	0	58
III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Low Tensile Strength Carbon-Steel Plates of Structural Quality for Welding (A 78 – 40)	103	0	54
Spec. for Low-Tensile Strength Carbon-Steel Plate of Flange and Firebox Qualities (A 89 – 39)	102	0	55
Spec. for Carbon-Silicon Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 39)	96	0	61

The recommendations in this report have been submitted to letter ballot of the committee which consists of 242 members, with the results shown in Table I.

TENTATIVE STANDARD APPROVED DURING THE PAST YEAR

On the recommendation of Committee A-1, Tentative Specifications for Heat-Treated Carbon-Steel Bolting Material (A 261 – 43 T) were approved[1] on April 27, 1943, by Committee E-10 on Standards. These specifications were developed in the Section on Bolting of Subcommittee XXII on Valves, Fittings, Piping and Flanges for High-Temperature Service in order to afford relief to many users of high-strength bolting material who were not able to procure the types of alloy steels which had been widely used. The specifications cover material up to 2 in. in thickness with a definite high ratio of yield point to tensile strength. The specifications are patterned after the widely used Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures (A 193 – 40 T).

WITHDRAWAL OF STANDARD SPECIFICATIONS

For reasons outlined later in this report (see discussion on emergency alternate provisions in still tubes and heat exchanger and condenser tube specifications) the following two standards were withdrawn:

Specifications for Seamless Cold-Drawn Alloy Steel (4 to 6 per cent Chromium) Heat-Exchanger and Condenser Tubes (A 187 – 40)

Specifications for Seamless Alloy-Steel (4 to 6 per cent Chromium) Still Tubes for Refinery Service (A 188 – 40)

This action was approved[2] by Committee E-10 on August 24, 1942. Actually certain of the grades in these standards are being retained in other specifications as emergency grades.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee A-1 reported the following results of the letter ballot of a total 159 ballots returned from a committee membership of 230: affirmative 111, negative 2, ballots marked "not voting" 46.

[2] In submitting these recommendations to Committee E-10 on Standards, Committee A-1 reported the following results of the letter ballot of a total of 166 ballots returned from a committee membership of 230: affirmative 90, negative 0, ballots marked "not voting" 76.

WORK OF NATIONAL EMERGENCY STEEL SPECIFICATIONS PROJECT

As a preface to a discussion of the several new emergency specifications developed during the year and the large number of emergency alternate provisions, it is desirable to comment briefly on a project in the War Production Board in which a large number of members of Committee A-1 on Steel have been extremely active, namely, the National Emergency Steel Specification Project (NESS). Based on the work of several NESS Technical Advisory Committees, the War Production Board issued a general Limitation Order L-211 prohibiting the production, fabrication, delivery, acceptance or use of any material except as covered in the schedules which from time to time will be appended to the order. A number of such schedules have been issued incorporating the recommendations of the various technical advisory committees to the Administrative Committee, in charge of the project, on which the Society is represented by N. L. Mochel, Chairman of Committee A-1, with Jerome Strauss, Chairman of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, serving as alternate. A list of the schedule numbers approved up to May 15 follows:

Schedules to Limitation Order L-211

No. of Schedule	Products Covered	Date of Issue
1	Concrete Reinforcement Steel	Oct. 23, 1942
2	Steel Wheels and Tires	Oct. 23, 1942
3	Barbed Wire, Wire Fence, Poultry Netting and Poultry Flooring	Nov. 12, 1942
	Amended	May 10, 1943
4	Structural Steel Shapes	Feb. 25, 1943
5	Steel Axles and Forgings (Railroad and Transit Services)	Feb. 25, 1943
6	Mechanical Steel Tubing	Feb. 25, 1943
7	Rails and Track Accessories	Feb. 25, 1943
	Amended	May 6, 1943
8	Carbon Steel Plates	Mar. 22, 1943
9	Oil Country Tubular Goods	Apr. 30, 1943
10	Water Well Tubular Products	Apr. 30, 1943

With the exception of Schedules 3, 9, and 10, all of the original schedules have been published in the ASTM BULLETIN. It is planned to reprint Nos. 9 and 10 in the August BULLETIN.

In selecting specifications for inclusion in the schedules, naturally many of the A.S.T.M. Standards developed by Committee A-1 on Steel have been included, and in a number of cases the technical advisory committees in order to aid in stimulating production or to conserve critical or strategic material have recommended that A.S.T.M. establish emergency alternate provisions. All of these have been promptly considered by the A-1 subcommittees in charge of the respective specifications. A number of the provisions discussed in the following section of the report are based on NESS proposals.

This report would not be complete unless it included a note of commendation to the large number of members of the Committee A-1 and of the chairmen of several of its subcommittees and officers for the splendid support given this NESS work.

COMPLETE EMERGENCY SPECIFICATIONS AND REVISIONS

Seven complete emergency specifications were accepted by Committee E-10 on January 30, 1943, following favorable letter ballot vote in Subcommittee VI on Forgings and the approval of the Subcommittee chairman, J. J. Laudig, and N. L. Mochel, chairman of Committee A-1. A list of these specifications follows:

ES - 21	Carbon-Steel and Alloy-Steel Forgings for Magnetic Retaining Rings for Turbine Generators
ES - 22	Alloy-Steel Forgings for Nonmagnetic Coil Retaining Rings for Turbine Generators
ES - 23	Carbon-Steel Forgings for Rings for Main Reduction Gears
ES - 24	Carbon-Steel and Alloy-Steel Forgings for Pinions for Main Reduction Gears
ES - 25	Carbon-Steel and Alloy-Steel Forgings for Turbine Generator Rotors and Shafts

ES - 26 Carbon-Steel and Alloy-Steel Forgings for Turbine Rotors and Shafts
ES - 27 Carbon-Steel and Alloy-Steel Forgings for Turbine Bucket Wheels

Tremendous demands for various types of heavy forgings, ship shafts, turbine parts, chemical equipment and so forth, caused particularly by naval and shipbuilding demands, brought rapidly to the forefront in May and June of 1942 the urgent desirability of standardizing specification requirements—the main objective being to stimulate increased production both in the ingot phase and through the forging and finishing processes. With the National Emergency Steel Specifications (NESS) project in force, the Administrative Committee agreed to the development of a Technical Advisory Committee on Heavy Forgings. C. J. Boyle, General Electric Co. (Chairman) and other metallurgists and technical men, practically all of whom are active members of the Society, have participated in this work. Steel producers, forging manufacturers, turbine and gear manufacturers and maritime interests including the U. S. Navy were represented.

Each of these specifications pertaining to various turbine and generator parts raised numerous questions which were reconciled through the close cooperation of those on the committee. One of the perplexing problems was to simplify chemical compositions. There were quite a large number of grades in use and of course to expedite tonnage and simplify steel making, forging and fabricating problems a limited number would be desirable. This achievement in the specifications represents an outstanding accomplishment. In order most effectively to achieve conservation of critical elements, chemical ranges are set up for some of the products—*exact* figures subject to agreement between producer and consumer. These turbine parts so extremely important in ships and other work in many cases need considerable alloying elements to guarantee against excessive wear and to produce the required combination of strength and ductility.

Revision of Emergency Specifications for Carbon-Chromium, Ball and Roller-Bearing Steels ES - 5a:

These emergency specifications developed in one of the NESS groups and subsequently approved through a special subcommittee of Committee A-1 were revised on September 16, 1942, to permit somewhat higher residuals of nickel and molybdenum, and requirements on decarburization and surface defects for ball wire and rods for cold heading were brought in line with current practice.

EMERGENCY ALTERNATE PROVISIONS

Some of the underlying reasons for the emergency provisions are discussed below, but in all cases they were established with two objectives in mind—first, increasing production of the steel or particular steel products covered; second, the conservation of critical and strategic alloying elements. Even though some of the changes may seem of a minor nature they nevertheless are important and entirely justified in the opinion of the committee.

Railroad and General Purpose Forgings:

The emergency provisions in the specification for carbon-steel axles, EA - A 21, increase availability of acid open-hearth steel by permitting a maximum phosphorus of 0.055 for acid steel and 0.05 for basic steel.

The provisions applying to the carbon-steel forgings for general industrial use, EA – A 235, established Class A as a carburizing steel providing a definite carbon content and deleting any physical requirements for this class and provides for heavy forgings over 20 in. in solid diameter or thickness.

The provisions in the alloy-steel forgings, EA – A 237, also established physical requirements for Class B forgings of large size.

The rather extensive provisions in the specification for carbon-steel forgings

TABLE II.—LIST OF EMERGENCY ALTERNATE PROVISIONS.
June 22, 1942 through June 1, 1943

Designation	Standard and Tentative Standard Specifications for:	Issued
FORGINGS		
EA – A 21	Carbon-Steel Axles for Cars and Tenders (A 21 – 36)	Oct. 5, 1942
EA – A 235	Carbon-Steel Forgings for General Industrial Use (A 235 – 42)	Jan. 30, 1943
EA – A 236	Carbon-Steel Forgings for Locomotives and Cars (A 236 – 42)	Aug. 12, 1942
EA – A 236a	" " " " " " " "	May 8, 1943
EA – A 237	Alloy-Steel Forgings for General Industrial Use (A 237 – 42)	Jan. 30, 1943
EA – A 238	Alloy-Steel Forgings for Locomotives and Cars (A 238 – 42)	Oct. 5, 1942
WHEELS		
EA – A 25	Wrought Steel Wheels for Electric Railway Service (A 25 – 41)	June 22, 1942
EA – A 25a	" " " " " " "	Aug. 24, 1942
EA – A 57	Multiple-Wear Wrought Steel Wheels (A 57 – 39)	June 22, 1942
EA – A 186	One-Wear and Two-Wear Wrought Steel Wheels (A 186 – 39)	June 22, 1942
EA – A 244	Heat-Treated Wrought Steel Wheels (A 244 – 42)	June 22, 1942
CASTINGS		
EA – A 148a	Alloy-Steel Castings for Structural Purposes (A 148 – 42)	April 27, 1943
PIPE AND TUBING		
Boiler and Superheater Tubes		
EA – A 83	Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 42)	Aug. 18, 1942
EA – A 178	Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 40)	Aug. 18, 1942
EA – A 192	Seamless Steel Boiler Tubes for High-Pressure Service (A 192 – 40)	Aug. 18, 1942
EA – A 209	Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 – 42)	Aug. 18, 1942
EA – A 213	Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 42)	Aug. 24, 1942
EA – A 226	Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 40)	Aug. 18, 1942
EA – A 249	Atomic-Hydrogen-Arc-Welded and Electric-Resistance-Welded Alloy-Steel Boiler and Superheater Tubes (A 249 – 42)	Aug. 24, 1942
EA – A 250	Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 41 T)	Aug. 24, 1942
Still Tubes for Refinery Service		
EA – A 161	Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 – 40)	Aug. 18, 1942
EA – A 200	Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 40)	Aug. 18, 1942
Heat-Exchanger and Condenser Tubes		
EA – A 179	Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 42)	Aug. 18, 1942
EA – A 199	Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 40)	Aug. 18, 1942
EA – A 214	Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 42)	Aug. 18, 1942
Pipe		
EA – A 53	Welded and Seamless Steel Pipe (A 53 – 42)	Jan. 30, 1943
EA – A 120	Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 42)	Aug. 24, 1942
EA – A 134	Electric-Fusion-Welded Steel Pipe (Sizes 30 in. and Over) (A 134 – 42)	Aug. 18, 1942
EA – A 135	Electric-Resistance-Welded Steel Pipe (A 135 – 42)	Aug. 24, 1942
EA – A 139	Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 – 42)	Aug. 18, 1942
EA – A 139a	Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 – 42)	April 8, 1943
EA – A 158	Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 – 42 T)	June 1, 1943
EA – A 206	Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 42 T)	June 1, 1943
EA – A 211	Spiral-Welded Steel or Iron Pipe (A 211 – 40)	Aug. 18, 1942

for locomotives and cars, EA - A 236, provide for use of Class A as a carburizing steel, modify phosphorus and sulfur content of certain classes and provide two considerably higher strength classes of materials in quenched-and-tempered condition designated H and I. These higher strength forgings were considered very essential by a number of the railroads so that it would not be necessary for them to redesign and rebalance certain parts which perhaps could not be obtained in alloy steels. Other changes were made to expedite production. EA - A 236a merely extends the scope of emergency Class H material to be applicable for locomotive and tender axles.

In EA - A 238 for alloy-steel forgings for locomotives and cars, phosphorus and sulfur content of acid open-hearth steel were raised.

Steel Wheels:

The emergency provisions in the four specifications listed in Table I permit sulfur content of acid steel to be as high as 0.06 per cent as compared with the present basic steel top of 0.05 per cent. The additional provisions in the wheels for electric railway service give requirements for spun steel wheels and as a service include the tables of dimensions of standard wheel designs part of the WPB Limitation Order L-211.

Steel Castings:

Emergency alternate provisions, EA - A 148a, for alloy-steel castings for structural purposes were extablished to concentrate production of six classes, which a very extensive survey of the industry indicated could be produced in reasonably large quantities. Two of the so-called regular grades, 1 and 2 of Class A, were retained and the other four are emergency grades, one of which, although now slightly modified, had been set up as an emergency on February 24, 1942.

Pipe and Tubing:

In this field all the emergency alternate provisions listed in Table II were developed in the NESS Technical Advisory Committee on Tubular Steel Products (T. G. Stitt, chairman); subdivided into six sections. Each section and the main committee include a large number of A-1 members, both consumers and producers. All of these provisions were approved in Subcommittee IX.

It is appropriate to refer to one phase of this work as an outstanding standardization achievement, namely, the establishment of a master list of some 18 different compositions of carbon and alloy steels for pressure tubes, which are a considerable reduction from existing standard grades, and then by use of emergency alternate provisions making it possible for production to be concentrated on steels with these chemical requirements. Primarily affected were tubular products in the boiler and superheater tubes field, still tubes, and heat exchanger tubes. A number of revised chemical requirements were justified to keep analyses in line with the scrap situation, others are intended to conserve molybdenum and other elements.

Discussion on Boiler and Superheater Tubes (see Table II).—The inclusion of "A.S.T.M. Specification Number" in the marking clause in A 83 and A 178 results from the desire of the Boiler Code Committee that this number be given and although there has been considerable objection to this on the part of the manufacturers, they have expressed willingness to go along with this change during this period as an emergency. Changes in the chemical requirements of Specifications A 192, A 209, A 226, A 249, and A 250 will bring the classes in line with emergency compositions being established. Waiving of the requirement to identify material as electric-resistance welded in marking clauses in Specifications A 226 and A 250 will help expedite production somewhat.

In Specifications A 213, a widely used standard, two new emergency classes TE 7 and TE 9 complete a series of grades by steps and enable the producer and consumer to effect most easily conservation of alloying elements.

Discussion on Still Tubes (*see Table II*).—As previously indicated, the change in molybdenum requirements is to conserve this very critical alloy. The transfer of the chromium-molybdenum type from Specifications A 188 to Specifications A 200 concentrates requirements for alloy-steel tubes in the one specification which will have six grades which the producers and consumers agree are essential. With the two remaining grades in Specifications A 188 obsolete for various reasons, primarily because of lack of use, there was no reason for continuing the specifications in A.S.T.M., and as previously indicated they have been withdrawn.

Minor changes in molybdenum content in Specifications A 161 have been made affecting the carbon-molybdenum grade which will conserve this element. The other changes in chemical requirements including those for silicon and sulfur should aid production.

Discussion on Heat Exchanger and Condenser Tubes (*see Table II*).—The notes on hydrostatic test requirements added in Specifications A 179 and A 214 follow the pattern used in other A.S.T.M. steel specifications providing for considerably higher hydrostatic testing when the material is to be used under more rigorous conditions than normally would be the case. Material conforming to these two specifications is used in all new extraction feedwater heaters, which operate at pressures considerably above 1000 psi.

Concerning the withdrawal of Specifications A 187 and the shift of its chromium-molybdenum grade to Specifications A 199, exactly the same situation holds as explained above pertaining to Specifications A 188 and A 200.

Discussion on Pipe Specifications (*see Table II*).—Certain of the emergency provisions in the seven specifications in this group are to make them more suitable during the emergency for use for water main pipe and water service. These specifications are being recommended to the WPB for incorporation in the list of so-called master steel specifications. The provisions concerning wall thickness were a distinct compromise between two quite different practices with respect to pipe thicknesses, namely, the use in the western United States and Pacific Coast area with its very light walls, and the Eastern practice, where considerably heavier walls are employed. For similar reasons references are being added to corrosion problems and coatings, and reference is to be made to the American Water Works Association requirements on protective coatings.

Specifications A 139 are widely used in connection with oil pipe lines, and the note being added in Section 14 of EA - A 139 referring to API thread standards is very important. The emergency provisions in EA - A 139a were set up to cover pipe sizes down to 4 in. in diameter (present size limited to 8 in. but not including 30 in.) and also to bring the requirements on elongation values in line with changes made as a regular feature in the widely used Specifications A 53 and A 106—these elongation values being set up according to wall thickness of the material.

The provisions in EA - A 53 were desirable in order to provide standardized requirements for a Grade C material which is used for water well pipe and is incorporated in Schedule 10 of WPB Order L-211. The NESS Technical Advisory Committee in recommending Specifications A 53 could not otherwise include Grade C and to simplify the mechanics this provision was established.

The provisions in EA - A 158 and EA - A 206 were provided in the interest of conservation of critical alloying materials, in particular molybdenum.

ACTIVITIES OF SUBCOMMITTEES

While several of the subcommittees have certain problems confronting them with respect to the status of specifications or decisions to cover the various products by standards, perhaps the only item which needs formal record in this report is the question of converter steel for castings. This subject has been discussed extensively. At the June, 1942, meeting in Subcommittee XXII on Valves, Fittings, Piping and Flanges for High-Temperature Service, a committee was appointed headed by V. T. Malcolm, with Sabin Crocker, and James Ferguson (alternate for L. C. Wilson). This committee transmitted a report based on a rather extensive survey of companies concerned with use of valves, flanges and fittings for high-temperature service. The committee concluded unanimously to recommend

> "That the A.S.T.M. casting specifications with regard to the process clause shall not be changed or modified."

This report subsequently was approved by Subcommittee XXII and by Committee A-1 at its January, 1943, meeting.

ACTING SUBCOMMITTEE CHAIRMEN

The chairmen of two subcommittees have suffered severe illnesses during the year.—E. F. Kenney, Bethlehem Steel Co., Chairman of Subcommittee I on Steel Rails and Accessories and also Vice-Chairman of Committee A-1; and T. G. Stitt, Pittsburgh Steel Co., Chairman of Subcommittee IX on Steel Tubing and Pipe and Chairman of the NESS Technical Advisory Committee on Tubular Steel Products. H. B. Oatley of the Superheater Co. has consented to serve as Acting Chairman of Subcommittee IX.

This report has been submitted to letter ballot of the committee which consists of 242 members; 150 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

N. L. MOCHEL,
Chairman.

H. P. BIGLER,
Secretary.
R. J. PAINTER
Staff Assistant.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-1 recommended to Committee E-10 on Standards that the Emergency Alternate Provisions in the Standard Specifications for Hard-Drawn Steel Spring Wire (EA - A 227), for Oil-Tempered Steel Spring Wire (EA - A 229), and for Carbon-Steel Valve Spring Quality Wire (EA - A 230) be withdrawn. This recommendation was accepted by Committee E-10 on July 6, 1943.

On August 30, 1943, Committee E-10 accepted additional Emergency Alternate Provisions in the Standard Specifications for Electric-Resistance-

Welded Steel Heat-Exchanger and Condenser Tubes (EA – A 214a) as well as Emergency Alternate Provisions in the Standard Specifications for Carbon-Steel Plates for Stationary Boilers and Other Pressure Vessels (EA – A 70) and for Low Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (EA – A 89.)

New Specifications for Carbon-Steel Seamless Drum Forgings, submitted by Committee A-1, were accepted by Committee E-10 for publication as tentative on November 17, 1943, and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 165, bearing the A.S.T.M. serial designation A 266 – 43 T.

On November 17, 1943, the following Emergency Alternate Provisions, submitted by Committee A-1, were accepted by Committee E-10:

Designation	Emergency Alternate Provisions In *Standard Specifications for:*
EA – A 30	Boiler and Firebox Steel for Locomotives (A 30 – 42)
EA – A 70	Carbon-Steel Plates for Stationary Boilers and Other Pressure Vessels (A 70 – 42)
EA – A 201	Carbon-Silicon Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 43)
EA – A 202	Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels (A 202 – 39)
EA – A 203	Low-Carbon Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 42)
EA – A 204	Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 204 – 42)
EA – A 212	High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (Plates 4½ in. and Under in Thickness) (A 212 – 39)

These emergency provisions were published in ASTM BULLETIN, No. 125, December, 1943, p. 64, and have also been issued in the form of pink stickers for attachment to the standards to which they apply.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

In this Appendix are given recommendations affecting certain standards and tentative standards on steel which are referred to earlier in the report.[1] The standards and tentative standards appear in their present form in the 1942 Book of A.S.T.M. Standards, Part I.

I. Revision of Tentative Standard

Tentative Specifications for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 – 42 T):

Section 2 (a).—Change from its present form to read as follows:

> *a)* The steel for lap-welded pipe shall be of good weldable quality made by the open-hearth process. The steel for seamless pipe grade A, and grade B (silicon-killed), shall be killed steel made by one or more of the following processes: open-hearth or electric furnace. The steel for seamless pipe grade B (bessemer) shall be killed steel made by the deoxidized acid-bessemer process.

Section 3.—Revise the requirements as to chemical composition by changing Table I from its present form, omitting the present footnote, to read as shown in the accompanying Table I.

Section 17.—Delete the words "seamless bessemer A" which appear in the first sentence in parentheses.

II. Revisions of Standards, Immediate Adoption

Standard Specifications for Low Tensile Strength Carbon-Steel Plates of Structural Quality for Welding (A 78 – 40):

Section 3 (a).—Change the manganese content of grades A and B from "0.35 to 0.60" to read "0.35 to 0.80" per cent.

Standard Specifications for Low Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (A 89 – 39):

Section 3 (a).—Revise the chemical composition requirements by changing the manganese content on all four grades in Table I from "0.35 to 0.60" to read "0.80 max." per cent.

TABLE I.—CHEMICAL REQUIREMENTS.

	Welded Open-Hearth	Seamless		
		Grade A	Grade B Silicon-Killed	Grade B Killed Acid-Bessemer
Carbon, max., per cent	....	0.25	0.35	0.25
Manganese, per cent	0.30 to 0.60	0.30 to 0.90	0.35 to 1.00	0.35 to 1.00
Phosphorus, max., per cent	0.045	0.04	0.04	0.11
Sulfur, max., per cent	0.06	0.06	0.06	0.06
Silicon, min., per cent	....	....	0.10	0.10

Standard Specifications for Carbon-Silicon Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 39):

Section 1.—Change the second sentence from its present form to read as follows:

> The maximum thickness of flange quality plates to be specified under these specifications

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[1] See p. 57.

shall be 2 in.; of ordinary firebox quality plates of grade A shall be 12 in.; and of grade B shall be 6 in.

Section 4.—Add under carbon, max., per cent for grade A the following:

	Grade A
For plates over 6 to 12 in., incl., in thickness	0.35

Section 8 (b).—At the end of Paragraph (*b*) add the following sentence:

The minimum elongation for grade A material shall be 15 per cent.

Section 9.—Change the table to read as follows:

Thickness of Material	Ratio of Pin Diameter to Thickness of Specimen	
	Grade A	Grade B
1 in. and under	½	1
Over 1 to 1½ in., incl.	1	1½
Over 1½ to 3 in., incl.	1½	2
Over 3 to 4 in., incl.	2	2½
Over 4 to 6 in., incl.	2½	3
Over 6 to 9 in., incl.	3	...
Over 9 to 12 in., incl.	3½	...

Section 10 (a).—Change the second sentence from its present form to read as follows:

The fractured surface of the test specimen shall not show any single seam or cavity more than ¼ in. in length for plates ¾ in. and under in thickness; nor more than ⅜ in. in length for plates over ¾ to 4 in., inclusive, in thickness; nor more than ½ in. in length for plates 4 to 6 in., inclusive, in thickness; nor more than ¾ in. in length for plates 6 to 12 in., inclusive, in thickness, in any of the fractures obtained in accordance with Paragraph (*b*).

Section 10 (b).—At the end of Paragraph (*b*) add the following sentence:

Where the press will not break the large specimen, the specimen may be split with a gas flame or by machining, and each section broken and examined.

Table II.—Change from its present form to read as shown in the accompanying Table II.

III. EDITORIAL CHANGES IN STANDARDS

Standard Specifications for Steel for Bridges and Buildings (A 7 – 42):

Section 5 (b).—Change from its present form to read as follows:

(*b*) Steel for plates and shapes 7/16 in. and under in thickness, and bars (other than those for rivets) 7/16 and under in thickness or diameter, intended for use in buildings and other structures subject to static loads only, may be made by the acid-bessemer process, unless otherwise specified.

TABLE II.—PERMISSIBLE VARIATIONS OVER ORDERED THICKNESS OF PLATES OVER 2 IN. IN THICKNESS.

Specified Thickness, in.	Variations over Specified Thickness for Widths Given					
	Up to 36 in., excl.	36 to 60 in., excl.	60 to 84 in., excl.	84 to 120 in., excl.	120 to 132 in., excl.	132 in. or over
Over 2 to 3, excl.	1/16	3/32	7/64	1/8	1/8	9/64
3 to 4, excl.	5/64	3/32	7/64	1/8	1/8	9/64
4 to 6, excl.	3/32	1/8	9/64	9/64	5/32	11/64
6 to 8, excl.	7/64	1/8	5/32	5/32	11/64	...
8 to 10, excl.	...	11/64	3/16	3/16	...	...
10 to 12, incl.	...	3/16	15/64	15/64	...	...

Standard Specifications for Welded and Seamless Steel Pipe (A 53 – 42):

Table I.—Round-off the decimal thicknesses to three places.

Section 15 (c).—Change from its present form to read as follows:

(*c*) *Couplings.*—Each length of threaded pipe shall be provided with one coupling, the threads of which shall be in accordance with the American Standard for Pipe Threads.[a] Threads shall be cut so as to make a tight joint. Couplings may be of wrought iron or steel. Taper tapped couplings shall be furnished on all weights of threaded pipe 2½ in. and larger.[b]

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[a] A complete description of the American Standard Pipe Threads applicable to pipe, valves, and fittings is contained in the American Standard for Pipe Threads (ASA No.: B2.1-1942); also "Screw-Thread Standards for Federal Services, 1942," National Bureau of Standards *Handbook H 28*, January, 1942, the pertinent data in both sources being identical.

[b] For sizes 2 in. and smaller, it is commercial practice to furnish straight-tapped couplings for standard-weight (Schedule 40) pipe and taper-tapped couplings for extra-strong (Schedule 80) and double-extra-strong pipe. If taper-tapped couplings are required for sizes 2 in. and smaller on standard weight (Schedule 40) pipe, line pipe in accordance with Specification 5L of the American Petroleum Institute should be ordered, thread lengths to be in accordance with the American Standard for Pipe Threads (ASA No. B2.1-1942). The taper-tapped couplings provided on line pipe in these sizes may be used on mill-threaded standard weight pipe of the same size.

Standard Specifications for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Pipe for Ordinary Uses (A 120 – 42):

Section 13 (*c*).—Change to read the same as recommended above for Section 15 (*c*) of Specifications A 53.

Standard Specifications for Electric-Resistance-Welded Steel Pipe (A 135 – 42):

Section 14 (*c*).—Change to read the same as recommended above for Section 15 (*c*) of Specifications A 53.

Standard Specifications for Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 – 42):

Section 14 (*c*).—Change from its present form to read as follows:

> (*c*) *Couplings.*—Each length of threaded pipe shall be provided with one taper-tapped coupling, having clean-cut threads of such a pitch diameter as to make a tight joint.

Tentative Specifications for Welded Alloyed Open-Hearth Iron Pipe (A 253 – 42 T):

Section 15 (*c*).—Change to read the same as recommended above for Section 15 (*c*) of Specifications A 53.

REPORT OF COMMITTEE A-3

ON

CAST IRON

Since the 1942 annual meeting of the Society in Atlantic City, N. J., Committee A-3 on Cast Iron has held one meeting, in St. Louis, Mo., on April 29, 1943. Another meeting will be held in June during the annual meeting of the Society.

During the year, 2 new members have been elected, and 1 resignation accepted; the total membership of the committee is now 118, of whom 38 are classified as producers, 47 as consumers, and 33 as general interest members.

Subsequent to the 1942 annual meeting, Committee A-3 presented to the Society through Committee E-10 on Standards a new Recommended Practice for Torsion Tests of Cast Iron. This recommended practice was accepted[1] by Committee E-10 for publication as tentative on August 24, 1942, and appears in the 1942 Book of A.S.T.M. Standards, Part I, bearing the A.S.T.M. serial designation A 260 – 42 T.

Committee A-3 has made an intensive effort to clarify and bring up to date all of its specifications in order that they would be of maximum utility in the war effort. This work has been accomplished satisfactorily, due to excellent cooperation of the membership.

Fortunately many years ago Committee A-3 evolved the policy of evaluating gray irons on the basis of physical test properties, with but few requirements as to chemical composition limitations. This policy has resulted in avoidance of the complex and confusing situation encountered in some other metals. Tremendous dislocation, and great effort has been involved where mechanical characteristic requirements have been accompanied by detailed composition requirements and complex chemistry. Under stress of war, and the scarcity of certain strategic materials, specifications for some metals have had to be altered almost continuously.

For fifteen years or more Committee A-3 has sought to promote the objective usage of cast iron on the part of engineers and designers. Despite some progress in that direction, war conditions have revealed that designers are not as yet fully cognizant of the possibilities inherent in the available classes of gray iron castings. To a considerable degree this condition is due to lethargy among the independent producers—the so-called jobbing foundries. It should be recognized that the production or tonnage of gray iron in the United States is approximately seven times the production of cast steel.

Analysis of the membership shows that the independent or jobbing shop interests are inadequately represented numerically. It is a policy of the Society to promote the intelligent usage of engineering materials by means of encouragement of research and the formulation of suitable specifications. Therefore the committee seeks to expand the direct interest of independent producers and producer groups, as well as consumers.

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee A-3 reported the results of the letter ballot vote as follows: Of a total membership of 115, 68 members returned their ballots, of whom 55 voted affirmatively, 0 negatively, and 13 members marked their ballots "not voting."

The magnaflux test is used to a limited extent in testing cast iron products. The committee would accordingly be interested in being represented should the Society undertake any work on this subject.

Committee A-3 has authorized the formation of a new subcommittee to review the properties of cast iron at elevated temperatures, with a view towards ultimately formulating specifications governing applications of cast iron at elevated temperatures. The new Subcommittee XXII on Elevated Temperature Properties of Cast Iron with J. S. Vanick, as chairman, may find it desirable to participate in, or to direct research activities necessary to establish clearly those properties and methods of test necessary for usage of cast iron as specified for elevated temperature applications.

The committee has recommended that the Society, as one of the sponsors of Sectional Committee A21 on Specifications for Cast-Iron Pipe and Fittings, endorse the submittal of the Threaded Cast Iron Pipe Specifications to the American Standards Association for approval as American Standard.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VII on Microstructure of Cast Iron (G. F. Comstock, chairman) held one meeting during the year, on March 3, 1943, in Buffalo, N. Y. The main topic discussed at this meeting was a proposal to amend the Tentative Recommended Practive for Evaluating the Microstructure of Graphite in Gray Iron (A 247 - 41 T) to include a new type of globular graphite discovered in an alloy cast iron at the National Bureau of Standards. Samples illustrating this type of graphite were distributed to some of those who attended the meeting, and the results of this study, although not yet entirely complete, do not indicate so far that a revision of the recommended practice is required.

Plans for a study of methods for determining cell size in gray cast iron, and for a study of nonmetallic inclusions in cast iron, especially in "inoculated" irons were also discussed at the meeting. Not much progress has been made yet along these lines, and future progress will very likely be slow until those who are interested in working on these subjects are less urgently occupied with important matters connected with the production of war materials.

Subcommittee XV on Impact Testing (J. T. Mackenzie, chairman) has before it the question of possible correlation of impact tests on cast iron and steel with regard to comparative numerical values, although the war prevents development of new testing machines and devices. Usage of cast iron under conditions where "toughness" is a recognized consideration will be investigated.

Subcommittee XXII on Elevated Temperature Properties of Cast Iron (J. S. Vanick, chairman) in cooperation with a committee of the American Foundrymen's Assn., has obtained the support of the War Metallurgical Committee of the Office of Scientific Research and Development. That group has practically completed an interesting and significant study of the actual field usages of gray iron under severe temperature conditions.

This report has been submitted to letter ballot of the committee, which consists of 117 members; 83 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. W. BOLTON,
Chairman.
J. T. MACKENZIE,
Vice-Chairman.

E. R. YOUNG,
Secretary.

REPORT OF COMMITTEE A-5

ON

CORROSION OF IRON AND STEEL

Committee A-5 on Corrosion of Iron and Steel held one meeting during the year, on June 22, 1942, in Atlantic City, N. J.

Since the last report 5 new members have been elected and 4 members have resigned or died. The committee membership now totals 131 of whom 59 are classified as producers, 44 as consumers, and 28 as general interest members.

RECOMMENDATIONS ACCEPTED BY COMMITTEE E-10 ON STANDARDS

Subsequent to the 1942 annual meeting, Committee A-5 presented to the Society through Committee E-10 on Standards a revision of the Tentative Specifications for Zinc Coating (Hot-Dip) on Hardware and Fastenings (A 153 - 33 T). The revised tentative specifications were accepted[1] by Committee E-10 on August 24, 1942, and appear in the 1942 Book of A.S.T.M. Standards, Part I.

I. REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption, the following revision in the Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Telephone and Telegraph Line Wire (A 111 - 33)[2] and accordingly asks for the necessary nine-tenths vote at the annual meeting in order that this modification may be referred to letter ballot of the Society:

Section 16 (*c*).—Change the first sentence to read as follows by the addition of the italicized figures and the omission of those in brackets:

> Each coil shall be securely bound in four separate places, equally spaced, with at least two wrappings of galvanized wire, not smaller than [0.109] *0.080* in. in diameter.

This change is proposed: (*1*) because the lighter binding wire is considered adequate, (*2*) because of the desire for conserving materials, and (*3*) because the application of a tight binding is troublesome when the 0.109-in. binding wire is used on coils of light gage wire now currently ordered.

II. TENTATIVE STANDARDS CONTINUED AS TENTATIVE WITHOUT CHANGE

No changes are being recommended in the other 13 specifications sponsored by the committee for reasons stated below under activities of Subcommittees VI and VII. As explained under the activities of Subcommittee VII, the Standard Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles (A 239 - 41) was clarified editorially in time for the corrected method to appear in the 1942 Book of A.S.T.M. Standards, Part I.

As explained under the activities of Subcommittee VI, no changes are being recommended in the tentative specifications which have stood for two years or more without revision.

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee A-5 reported results of the letter ballot vote as follows: Of a total membership of 130, 75 members returned their ballots, of whom 65 voted affirmatively, 0 negatively, and 10 members marked their ballots "not voting."

[2] 1942 Book of A.S.T.M. Standards, Part I, p. 509.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 131 members with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Item	Affirmative	Negative	Ballots Marked "Not Voting"
I. Revision of Standard, Immediate Adoption			
Spec. for Zinc-Coated (Galvanized) Iron or Steel Telephone and Telegraph Line Wire (A 111 - 33)	88	1	7
II. Tentative Standards Continued as Tentative Without Change			
Spec. for Zinc-Coated (Galvanized) Iron or Steel Sheets (A 93 – 38 T)	84	0	16
Spec. for Electrodeposited Coatings of Zinc on Steel (A 164 – 40 T)	82	0	18
Spec. for Electrodeposited Coatings of Cadmium on Steel (A 165 – 40 T)	78	0	22
Tests for Local Thickness of Electrodeposited Coatings (A 219 - 40 T)	52	0	13

Activities of Subcommittees

Subcommittee III on Inspection of Annapolis Tests (E. S. Taylerson, chairman).—The specimens of copper-bearing and noncopper-bearing corrugated black sheets that have been exposed at Annapolis, Md., since October 17, 1916, were inspected on April 28 and October 16, 1942. No failures were found at the April inspection. In October, two No. 22 gage sheets had failed (B8 and I6). No failures of No. 16 gage material were noted this year.

A summary of the failures to date is given in the accompanying Table II.

Subcommittee V on Total Immersion Tests (W. J. Jeffries, chairman).—It is necessary to defer for a year the final report of this committee because time has not been available to complete a statistical study of the large amount of accumulated data.

Subcommittee VI on Specifications for Metallic-Coated Products (T. R. Galloway, chairman).—The several specifications under the jurisdiction of this committee are remaining unchanged for the reasons noted below:

(*a*) No changes are deemed necessary. This applies to specifications for barbed wire, farm-field and railway right-of-way fencing, and strand.

(*b*) Restrictions on the use of materials make the products covered unavailable, such as chain-link fence galvanized after weaving.

(*c*) Other circumstances of war-time activities or the preoccupation of committee members with other work make the revision of existing standards impracticable or untimely. This applies to the specifications for electroplated coatings of zinc or cadmium on steel and the specifications for galvanized steel sheets.

Also because committee members are busy with other work, or because of scarcity of materials, certain projects are being delayed. This is the case with new specifications on chain-link fence galvanized before weaving and on terneplate specifications.

While none of the four tentative specifications for which Committee A-5 is responsible is being changed at this time, it is planned to adopt the Tentative Specifications for Electrodeposited Coatings of Zinc on Steel (A 164 – 40 T), and the Tentative Specifications for Electrodeposited Coatings of Cadmium on Steel (A 165 – 40 T) as standard as soon as Committee B-8 on Electrodeposited Metallic Coatings completes certain pertinent work on provisions dealing with sampling and inspection. The Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Sheets (A 93 – 38 T) are not being recommended for adoption as standard, because it has not been possible during

TABLE II.—RECORD OF FAILURES AT ANNAPOLIS OF 22 GAGE COPPER-BEARING AND NON-COPPER-BEARING CORRUGATED BLACK SHEETS.

Sheets Exposed October 17, 1916. Inspections Made Semiannually from 1917 to 1943.

Group	Designation	Series	Average Analysis, per cent						Number of Sheets in Test[a]	Life of Each Failed Sheet, yr.																Number of Sheets in Test	Number of Sheets Failed	Average Life of Sheets, yr.	Group
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper																					
Z......	Open-hearth Steel	B	0.032	0.32	0.063	0.042	..	0.019	3	13.5	17.0	19.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	16.5	.....Z
O.....	Open-hearth Steel	A	0.121	0.536	0.008	0.030	0.249	0.020	12	7.0	9.0	9.0	9.0	9.0	9.5	10.0	10.0	11.0	11.0	13.0	17.0	..	..	..	..	12	12	10.4	....O
B.....	Open-hearth Steel	A	0.09	0.038	0.017	0.062	0.036	0.084	3	20.0	20.5	24.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	21.5	....B
M.....	Open-hearth Steel	B	0.093	0.419	0.013	0.041	0.011	0.185	13	20.0	20.5	23.5	23.5	23.5	24.0	24.0	24.0	24.0	24.0	24.0	24.0	25.0	..	..	..	13	13	23.4	....M
B.....	Open-hearth Steel	A	0.06	0.27	0.014	0.053	0.006	0.187	9	19.5	20.0	21.5	22.0	22.5	23.0	26.0	*	*	..	..	..	..	..	..	..	9	7	..	....B
Z.....	Open-hearth Steel	B	0.067	0.351	0.045	0.052	0.003	0.187	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	.....Z
T.....	Open-hearth Steel	B	0.083	0.313	0.009	0.020	0.005	0.227	14	19.5	19.5	19.5	21.5	21.5	21.5	23.0	23.5	23.5	23.5	24.0	24.0	24.0	24.0	..	..	14	14	22.3	....T
K.....	Open-hearth Steel (Acid)	A	0.107	0.447	0.091	0.046	0.004	0.237	14	24.5	*	*	*	*	*	*	*	*	*	*	*	*	*	..	..	14	1	..	....K
H.....	Open-hearth Steel	A	0.069	0.387	0.016	0.027	0.004	0.244	10	19.5	21.0	21.5	23.0	23.5	23.5	23.5	24.0	24.0	25.0	..	..	..	..	..	..	10	10	22.9	....H
Z......	Open-hearth Steel	B	0.065	0.35	0.050	0.059	0.003	0.264	2	24.5	*	..	..	..	..	..	..	..	..	..	..	..	..	..	..	2	1	..	.....Z
Z......	Open-hearth Steel	B	0.073	0.42	0.052	0.054	Trace	0.641	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	.....Z
S.....	Open-hearth Iron	B	0.02	0.023	0.006	0.022	0.004	0.022	14	7.0	8.0	8.5	9.0	9.5	10.0	11.0	11.5	11.5	12.5	12.5	12.5	12.5	12.5	..	..	14	14	10.6	.....S
D.....	Open-hearth Iron	A	0.02	0.026	0.004	0.022	0.002	0.024	6	16.0	16.0	16.0	19.5	19.5	23.5	..	..	..	..	..	..	..	..	..	..	6	6	18.4	....D
X.....	Open-hearth Iron	B	0.021	0.055	0.007	0.031	0.004	0.027	14	10.5	13.0	13.5	14.5	14.5	16.0	16.0	16.5	16.5	17.0	17.5	17.5	18.0	19.0	..	..	14	14	15.7	X
C.....	Open-hearth Iron	A	0.01	0.028	0.002	0.027	0.002	0.141	3	17.5	20.5	23.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	20.5	C
C.....	Open-hearth Iron	A	0.015	0.028	0.006	0.036	0.003	0.194	9	16.5	16.5	18.5	19.0	19.0	20.0	20.5	21.5	21.5	..	..	..	..	..	..	..	9	9	19.2	C
U.....	Open-hearth Iron	B	0.017	0.074	0.008	0.037	0.005	0.260	13	15.5	16.5	19.0	19.0	19.0	19.0	19.0	19.5	20.0	20.0	21.0	21.0	22.5	..	..	..	13	13	19.3	....U
V.....	Open-hearth Iron	B	0.018	0.051	0.007	0.021	0.005	0.304	14	17.0	17.0	17.5	18.5	18.5	19.0	20.0	20.0	20.5	21.0	22.0	22.0	22.5	24.0	..	..	14	14	20.0	V
A.....	Bessemer Steel	A	0.038	0.386	0.089	0.040	0.007	0.014	15	15.5	16.5	16.5	17.0	17.0	17.0	18.0	19.0	19.0	19.5	19.5	19.5	19.5	19.5	20.0	..	15	15	18.2	....A
Z......	Bessemer Steel	B	0.063	0.41	0.102	0.044	0.009	0.013	3	11.5	11.5	11.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	11.5	.....Z
Z......	Bessemer Steel	B	0.077	0.44	0.095	0.036	0.003	0.140	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	.....Z
I......	Bessemer Steel	A	0.041	0.365	0.097	0.068	0.008	0.252	15	24.0	26.0	*	*	*	*	*	*	*	*	*	*	*	*	*	..	15	2	..	I
Z......	Bessemer Steel	B	0.058	0.37	0.093	0.043	0.009	0.269	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	.....Z
Z......	Bessemer Steel	B	0.055	0.38	0.110	0.046	..	0.527	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	.....Z
Y.....	Wrought Iron	B	0.03	0.055	0.139	0.021	0.218	0.020	3	18.0	19.5	23.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	20.3	....Y
E.....	Wrought Iron	A	0.033	0.034	0.114	0.021	0.134	0.283	10	19.5	20.5	21.0	21.0	23.0	23.0	23.0	23.0	24.0	25.0	..	..	..	..	..	..	10	10	22.3	....E
							Total........214																			Total...214	170		

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* Indicates unfailed sheet.

[a] The number of sheets in the test indicated in this table is less than the number shown in earlier reports because certain sheets were discarded due to mechanical damage.

this war-time period to carry on the needed work of correlating these specifications with the Federal specification covering galvanized sheets.

Subcommittee VII on Methods of Testing (R. W. Baker, chairman).—The Standard Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles (A 239 - 41) has been editorially revised to better define the end point of the test (Section 8 (*a*)). This change was completed in time to permit of its inclusion in the 1942 Book of A.S.T.M. Standards, Part I.

It is recommended that the Tentative Methods of Test for Local Thickness of Electrodeposited Coatings (A 219 - 40 T) which is under the joint jurisdiction of Committees A-5 and B-8 be continued as tentative without revision.

A Field Conformance Test Subgroup is investigating the limitations of the various field testing methods previously reported on (such as dropping test methods and magnetic test methods) for determining the thickness of protective coatings.

Subcommittee VIII on Field Tests of Metallic Coatings (R. F. Passano, chairman).—This subcommittee has continued the regular inspections of its large collection of uncoated sheet specimens and of coated sheet, wire, fence, strand, and hardware specimens undergoing atmospheric exposure tests, thus maintaining the continuity of these fact-finding studies even in war time.

One feature of this maintenance of continuity was the reconditioning of hardware racks at State College, Pa., and at Sandy Hook, N. J., and the remounting of such hardware specimens as appeared to merit further continuation in the test. The specimens remounted consisted mostly of those carrying hot-dip zinc, aluminum, or lead coatings, and some having sherardized coatings. The specimens carrying electroplated coatings (zinc or cadmium), and most of those which had been sherardized had become too rusty to salvage because their coatings were originally relatively light. Less than a quarter of the specimens were remounted at each location.

Tables III and IV, show the identification and positioning on the racks of the remounted samples as well as the positions they occupied on the original racks.

In line with the precedent for annual reports of this committee, detailed inspection records of coated sheets and hardware are omitted in odd numbered years because of the large volume of inspection records being published in this report on wire, fencing, and strand.

This report has been submitted to letter ballot of the committee, which consists of 131 members, 100 members returned their ballots, of whom 96 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. D. HOCKER,
Chairman.

J. B. DIXON,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-5 presented to the Society through Committee E-10 on Standards Emergency Alternate Provisions in the Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (A 116 - 39) and for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (A 121 - 39). These emergency alternate provisions were accepted by Committee E-10 on Standard on January 24, 1944, and appear in the ASTM BULLETIN, No. 127, March, 1944, and have been issued in the form of pink stickers for attachment to the standards.

TABLE III.—ARRANGEMENT OF HARDWARE SPECIMENS ON RACKS RECONSTRUCTED AT SANDY HOOK, N. J., ON OCTOBER 30, 1942.

Abbreviations denoting types of coating: HZ = hot-dip zinc; EZ = electroplated zinc; ES = electric sherardized; GS = gas sherardized; Pb = lead(or amaloy); Al = hot-dip aluminum; Cal = calorized.

Note.—g (applied to new specimen numbers) = specimens retrieved from ground and remounted.

[a] Old numbers denote specimen number and positions occupied in Old Bays.

[b] Groups of old numbers not individually correlated with new numbers.

BAY NO. I

(Most northerly bay on west side of racks)

	3 by 3 by 5/16-in. Angles			1½ by 1½ by ⅛-in. Angles		
	HZ	Al		HZ	Al	Pb
New numbers....................	1g 2g 3g 4g 5g 6g	7g 8g 9g 10g 11g 12g		22 23 24	25 26 27	28 29 30
Old numbers[a]....................	4, 5, 6, 34, 35, 36[b]	7, 8, 9, 37, 38, 39[b]		22 23 24	7 8 9	19 20 21
	Transferred from Old Bay No. I			Transferred from Old Bay No. V		

	2-in. Conduit				1½ by 1½ by ⅛-in. Angles		
	HZ	Al	Pb		HZ	Al	Pb
New numbers..................	13 14 15	16 17 18	19 20 21		31 32 33	34 35 36	37 38 39
Old numbers[a]..................	4 to 6[b]	7 to 9[b]	25 to 27[b]		37 38 39	40 41 42	31 32 33
	Transferred from Old Bay No. III				Transferred from Old Bay No. V		

BAY NO. II

(Most northerly bay on east side of racks)

(See Notes below for abbreviations (TF) (N & L) etc.)

New No.	Old No.	New No.	Old No.	New No.	Old No.	New No.	Old No.	New No.	Old No.
HZ	(N&L)	Al	(N&L)	HZ	(TC)	Al	(TC)	Pb	(KO)
1	49	10	58	17	118	28	160	36	28
2	50	11	59	18	119	29	161	37	29
3	51	12	60	19	120			38	30
				20	122	HZ	(KO)		
4	52			21	123	30	7	HZ	(BDCl)
5	53			22	124	31	8	39	4
6	54	Pb	(N&L)	23	125	32	9	40	5
		13	97	24	126			41	6
		14	98						
Al	(N&L)	15	99	Al	(TC)	Al	(KO)	Al	(BDCl)
7	55			25	157	33	31	42	22
8	56			26	158	34	32	43	23
9	57	16	100	27	159	35	33	44	24

	2 by 1½ by 5/16-in. Angles		
	HZ	Al	Pb
New Nos.	45g 46g 47g 48g 49g	50g 51g 52g 53g 54g 55g	56g 57g
Old Nos.[a]	Some 5 of 4, 5, 6, 37, 38, 39[b]	All of 7, 8, 9, 40, 41, 42[b]	Some 2 of 19, 20, 21, 52, 53, 54[b]
	Transferred from Old Bay No. II		

	No. 792 Window-Frame Section						No. 14 by 2½-in. Flats								
	HZ			Al			HZ			Al			Pb		
New Nos.	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72
Old Nos.[a]	37	38	39	40	41	42	7	8	9	10	11	12	22	23	24
	Transferred from Old Bay No. X						Transferred from Old Bay No. VI								

Specimens transferred from Old Bay No. IV. Numbers 1 to 16: Nipples & Ells (N&L). Numbers 17 to 29: Trolley Clamps (TC).
Specimens transferred from Old Bay No. VIII. Numbers 30 to 38: Knock out Boxes (KO). Numbers 39 to 44: Bull Dog Clamps (BDCl).

BAY NO. III

(Bay on West side of racks, adjacent to Bay No. I)

	Top Section Vacant																		
	¼ by 3-in. Steel Flats										¼ by 3-in. Steel Flats								
	HZ			Al			Pb				HZ			Al			Pb		
New numbers	1	2	3	4	5	6	7	8	9		10	11	12	13	14	15	16	17	18
Old numbers[a]	7	8	9	10	11	12	22	23	24		37	38	39	40	41	42	52	53	54
	Transferred from Old Bay No. IX										Transferred from Old Bay No. IX								

TABLE IV.—ARRANGEMENT OF HARDWARE SPECIMENS ON RACKS RECONSTRUCTED AT STATE COLLEGE, PA., ON NOVEMBER 16, 1942.

Abbreviations denoting types of coating: HZ = hot-dip zinc; EZ = electroplated zinc; ES = electric sherardized; GS = gas sherardized; Pb = lead (or amaloy); Al = hot-dip aluminum; Cal = calorized.

Note.—g (applied to new specimen numbers) = specimens retrieved from ground and remounted.

[a] Old numbers denote specimen number and positions occupied in Old Bays.

[b] Groups of old numbers not individually correlated with new numbers.

BAY NO. I

(Most northerly bay on west side of racks)

	3 by 3 by 5/16 in. Angles						
	HZ	Al	Pb	ES	HZ	Al	Pb
New numbers	1g 2g 3g	4g 5g 6g	7g 8g 9g	10g 11g 12g	13 14 15g	16 17 18	19 20
Old numbers[a]	4 to 6[b]	7 to 9[b]	16 to 18[b]	13 to 15[b]	34 35 36	37 38 39	46 47
	Transferred from Old Bay No. I						

	2-in. Conduit						1-in. Conduit		
	EZ	HZ	Al	GS	Pb	EZ	HZ	Al	ES
New numbers	21 22 23	24 25 26	27 28 29	30 31 32	33 34 35	36 37 38	39 40 41	42 43 44	45 46 47
Old numbers[a]	1 2 3	4 5 6	7 8 9	22 23 24	25 26 27	31 32 33	34 35 36	37 38 39	46 47 48
	Transferred from Old Bay No. III								

BAY NO. II

(Most northerly bay on east side of racks)

	2½ by 1½ by 3/16 in. Steel Angles		
	HZ	Al	Pb
New numbers	1 2 3	4 5 6	7 8 9
Old numbers	7 8 9	10 11 12	19 20 21
	Transferred from Old Bay No. II		

	No. 792 Window Frame Sections		No. 14 by 2½ in. Steel Flats			2½ by 1½ by 3/16 in. Steel Angles		
	HZ	Al	HZ	Al	Pb	HZ	Al	Pb
New numbers	10g 11g 12g	13g 14g 15g	16 17 18	19 20 21	22 23 24	25 26 27	28 29 30	31 32 33
Old numbers[a]	37 to 39[b]	40 to 42[b]	37 38 39	40 41 42	52 53 54	37 38 39	40 41 42	52 53 54
	Transferred from Old Bay No. X		Transferred from Old Bay No. VI			Transferred from Old Bay No. II		

BAY NO. III

(Bay on west side of racks, adjacent to Bay No. I)

	3 by 3-in. Angles		1½ by 1½ by ⅛-in. Angles				1¼ by 3-in. Flats		
	Pb	ES	HZ	Al	Pb		HZ	Al	Pb
New numbers	1	2 3 4	5 6 7	8 9 10	11 12 13		14 15 16	17 18 19	20 21 22
Old numbers[a]	48	43 44 45	7 8 9	10 11 12	22 23 24		7 8 9	10 11 12	22 23 24
	Transferred from Old Bay No. I		Transferred from Old Bay No. V				Transferred from Old Bay No. IX		

	1-in. Conduit				1½ by 1½-in. Angles			¼ by 3-in. Plate			
	GS	Pb	Pb	Cal	Zn	Al	Pb	EZ	HZ	Al	Pb
New numbers	23 24 25	26 27 28	29 30 31	32 33 34	35 36 37	38 39 40	41 42 43	44 45 46	47 48 49	50 51 52	53 54 55
Old numbers[a]	52 53 54	28 29 30	58 59 60	55 56 57	37 38 39	40 41 42	52 53 54	34 35 36	37 38 39	40 41 42	52 53 54
	Transferred from Old Bay No. III				Transferred From Old Bay No. V			Transferred from Old Bay No. IX			

BAY NO. IV

(Bay on east side of racks, adjacent to Bay No. II)

(See Notes below for abbreviations (TF) (N&L) etc.)

New No.	Old No.	New No.	Old No.	New No.	Old No.	New No.	Old No.	New No.	Old No.	New No.	Old No.	New No.	Old No.	New No.	Old No.	New No.	Old No.	
HZ	(TF)	ES	(TF)	EZ	(N&L)	HZ	(N&L)	ES	(N&L)	Al	(N&L)	HZ	(TC)	HZ	(KO)	Pb	(KO)	
1	7	7	19	13	37	24	49	33	92	41	58	51	123	60g	7	67g	30	
				14	38	25	50	34	93	42	59	52	124	61g	8			
		8	20	15	39	26	51			43	60	53	125	62g	9	ES	(KO)	
2	8											54	126					
				16	40			Pb	(N&L)							68g	22	
		9	21	17	41	27	52	35	97	HZ	(TC)			Al	(KO)			
						28	53									69g	23	
3	9	Al	(TF)	18	43			36	98	44	115	Al	(TC)					
						29	54							63g	32	70g	24	
		10	13	19	44			37	99	45	116	55	157					
														64g	33			
				20	45					46	117	56	159					
4	10					Al	(N&L)									Al	(BDCl)	
		11	14							47	118							
										48	120			Pb	(KO)			
5	11			21	46			38	100			57	160			71	22	
				22	47	30	55	39	101			58	161			72	23	
						31	56			49	121			65g	28			
6	12	12	15	23	48	32	57	40	102	50	122	59	162	66g	29	73	24	

Specimens Transferred from Old Bay No. IV Nos. 1 to 12: Tower Fittings (TF). Nos. 13 to 43: Nipples and Ells (N&L). Nos. 44 to 59: Trolley Clamps (TC).
Specimens Transferred from Old Bay No. VIII Nos. 60 to 70: Knockout Boxes (KO). Nos. 71 to 73: Bull Dog Clamps (BDCl).

REPORT OF WIRE INSPECTION COMMITTEE ON FIELD TESTS OF WIRE AND WIRE PRODUCTS

(COVERS 1941 AND 1942 INSPECTIONS)

NOTE.—Reference to the previously published reports of Subcommittee VIII on Field Tests of Metallic Coatings, of Committee A-5 on Corrosion of Iron and Steel, which appeared in the 1939 and 1941 *Proceedings*,[1] will be helpful in certain studies of this exposure test because it contains all the data assembled in inspections prior to 1941, descriptions of the test methods as well as micrographs and other characterizations of specimens in the test.

This report presents the results of the atmospheric corrosion tests on wire and wire product specimens after exposure for about six years at each of eleven locations. These eleven test sites have atmospheres of divers types, including severely industrial, mildly industrial, seacoast, and rural. At each location, more than 900 specimens were exposed. These included short lengths of wire (42 in. long) and wire strand at all locations; farm field fence at nine sites; barbed wire at eight locations; and chain-link fence at eight places. Previous reports show the details of the locations and of the number and nature of the test specimens involved.[2]

The extent of corrosions on specimens is being measured by the following:

1. Visual examination at the test locations either semiannually or annually, depending on the corrosiveness of the test site atmosphere. At the time of inspection, records are made of the appearance of every wire and wire product sample at each test site. The records are:

(*a*) Qualitative descriptions prior to the time of the appearance of rust or rust-resembling corrosion products; that is, the samples are noted as appearing "metallic," "gray," "brown," "black," etc.

(*b*) Quantitative estimations after the appearance of rust or rust-like corrosion products. Estimates are made of the percentage of area on each sample that has actually rusted and the percentage that has "yellowed" by iron corrosion products, but has not yet shown actual rusting of the base metal.

2. Tension tests on the unfabricated wire specimens. These are the specimens exposed in groups of seven comparison samples taken from the same lot of wire and supposedly alike. An attempt is made to remove and test the first specimen of each heptad when it has lost 5 to 10 per cent of its strength; the seventh sample, when it has lost some 75 per cent of its strength; and the intermediate five samples when they have lost suitable proportions of their strength.

The records of progressive loss of strength of the unfabricated wire samples will afford an indication of the rate of loss of strength of the wires in the farm fence and barbed wire specimens, because wire from nearly every lot used in the fabrication of the fences and

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 99 (1939), and Vol. 41, p. 101 (1941).

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 156 (1939).

TABLE A.—CORROSION CONDITIONS OF ZINC-COATED UNFABRICATED WIRES AFTER APPROXIMATELY SIX YEARS' EXPOSURE.

R = rust; Y = yellowed; G = gray; M = metallic; MG and GY = intermediate states.

Location	Coating Group, oz. per sq. ft. of surface										
	0.20 to 0.30	0.25 to 0.35	0.35 to 0.45	0.45 to 0.55	0.50 to 0.60	0.60 to 0.70	0.70 to 0.85	0.80 to 1.00	1.20 to 1.35	1.60 to 1.85	2.00 or more
Pittsburgh, Pa.	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	G
Sandy Hook, N. J.	100R	100R	100R	100R	60R	63R	31R	1R	GY	G	G
Bridgeport, Conn.	100R	100R	100R	100R	91R	58R	40R	3R	GY	G	G
State College, Pa.	62R	51R	1R	1R	GY	GY	GY	GY	G	G	G
Lafayette, Ind.	100R	75R	36R	1R	MG	MG	MG	MG	MG	MG	MG
Ames, Iowa	33R	21R	G	G	G	G	G	G	G	G	G
Manhattan, Kans.	G	G	G	G	MG	MG	MG	MG	MG	MG	MG
Ithaca, N. Y.	70R	38R	G	G	MG	MG	MG	MG	MG	MG	M
Santa Cruz, Calif.	G	G	G	G	MG	MG	MG	MG	....	MG	M
College Station, Tex.	2R	1R	GY	GY	GY	GY	GY	G	G	G	G
Davis, Calif.	G	G	G	G	MG	MG	MG	MG	....	MG	M

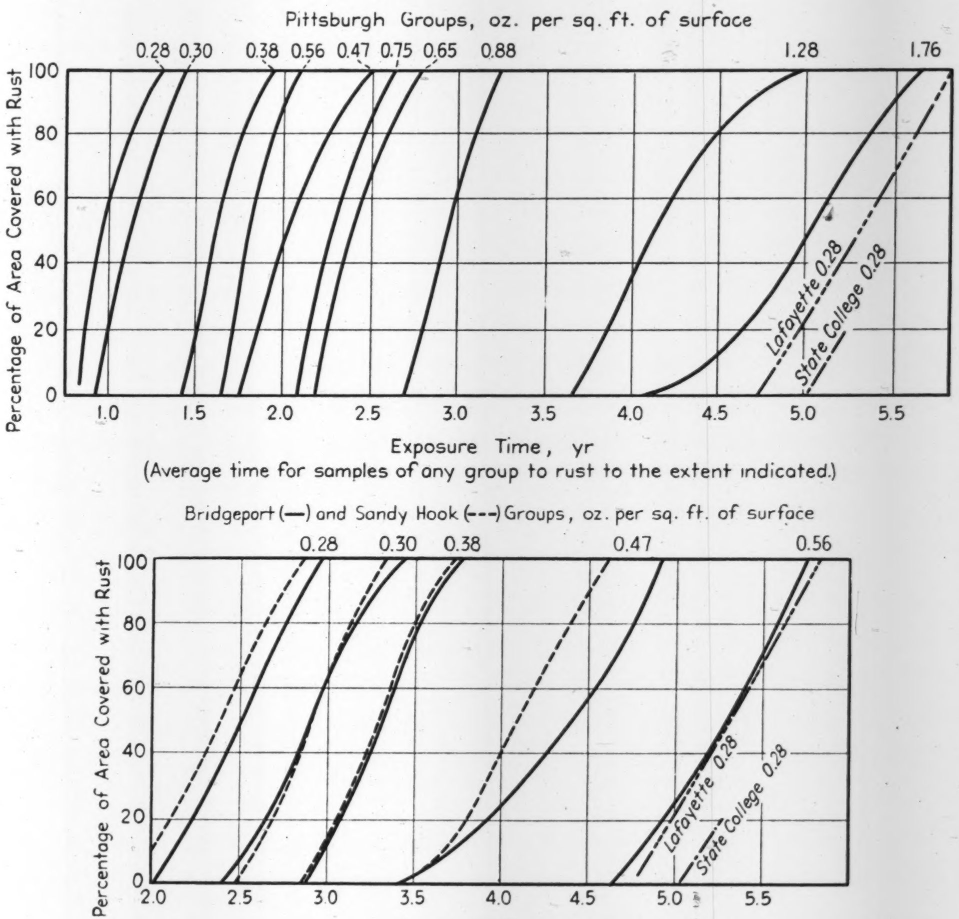

FIG. 1.—Progressive Development of Rust on Zinc-Coated Unfabricated Wire.

Coating weight groups in which all of the samples are rusted.

barbed wires is also under test as unfabricated wire.

3. Loss of weight tests are being made on lengths of zinc-coated wire (each about 42 in. long) carefully weighed and measured before exposure. There are 144 wires (16 alike of each of 9 wire lots) exposed at each of the locations of Pittsburgh, Sandy Hook, and State College. These wires originally carried weights of coating of 0.66 to 1.04 oz. per sq. ft. The working scheme for handling these specimens at each location is to remove one fourth at a time—the first quarter when they have lost a relatively small percentage of their coating; the last quarter when most of the coating has gone but before significant rusting has occurred; and the intermediate two one-quarter lots when they have lost suitable proportions of their coating.

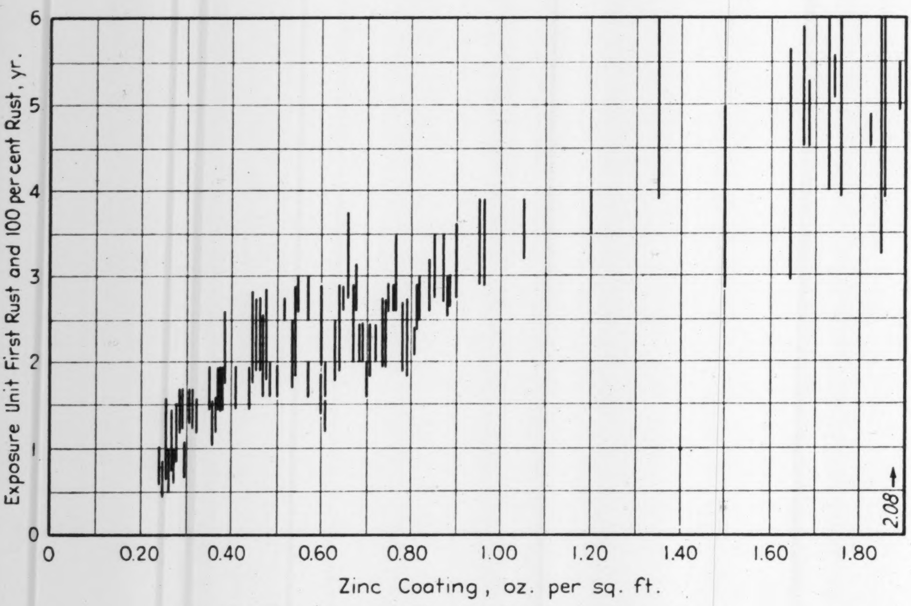

FIG. 2.—Exposure Time for Rusting to Occur.

Semiannual inspections have been made at Pittsburgh, Bridgeport, Sandy Hook, and State College by a traveling committee and annual inspections have been made at the other test sites by the university people in charge of the test plots.

Findings from Visual Examination for Corrosion at the Test Plots:

The detailed corrosion inspection records collected in 1941 and 1942 are presented in Table I (Plate I) for unfabricated wire specimens, Table II for barbed wire, Table III for farm-field fence, Table IV for chain-link fence, and Table V for wire strand.

These tables are similar in form to the tables in previous reports except that in the case of Table I for unfabricated wire specimens all the inspection data from Pittsburgh has been assembled together with a graphic presentation of the period of rusting.

Unfabricated Wire Specimens Carrying Zinc Coatings.—Table A summarizes the corrosion condition of the zinc-coated unfabricated wire specimens after exposure for about six years at each of the eleven test sites.

Figure 1 shows the progressive development of rust of all groups of wires which have become 100 per cent rusted and of all groups of wires which, while not 100 per cent rusted, show some rust on each specimen in the group.

Figure 2 shows the time for appearance of first rust and the time for complete rusting in relation to the weights of coating on all unfabricated wire specimens at Pittsburgh which have rusted.

Unfabricated Wire Specimens Carrying Coatings Other Than Zinc.—The copper covered wires show no rusting at any location. The uncoated corrosion-resistant steel wires show superficial corrosion or yellowing (often in a spotted pattern) at several locations. Generally the 18 per cent chromium, 8 per cent nickel content wires retain their original metallic luster longer than the 13 to 14 per cent chromium content wires.

Nearly all of the lead-coated wires show some pinhole rusting at all locations. Similar to the experience with hardware samples, there are various instances where the lead-coated wires appear less corroded now than they did at earlier inspections.

Barbed Wire and Farm-Field Fence.—These products are exposed at all locations except Pittsburgh, Sandy Hook, and Bridgeport. The barbed wire shows some rust in specimens in the 0.20 to 0.30 oz. per sq. ft. coating group at State College, Lafayette, Ames, and Ithaca, and on samples in the 0.35 to 0.45 oz. per sq. ft. coating group at State College, Lafayette and Ithaca. The lead-coated barbed wire specimens show some pinhole rust at most locations.

Rusting of farm-field fence specimens has started among the two lightest weight coating groups—0.20 to 0.30 and 0.25 to 0.35 oz. per sq. ft.—at State College, Lafayette, Ames, Ithaca, College Station, and Davis, and on specimens of the 0.35 to 0.45 oz. per sq. ft. coating group at State College, Lafayette, Ithaca, and College Station. Lead-coated fences generally show the pinhole rusting and yellowing characteristics of all lead-coated specimens in the test.

The corrosion performance of any individual specimen of barbed wire or of farm-field fence listed in Tables II and III may be directly compared with the performance of specific unfabricated wire specimens. The interior line and stay wires of every fence, the top and bottom line wires of every fence, and the line wires of every barbed wire specimen are made of wire of an identified lot number. These same lots of wire are usually under test as unfabricated wire. The Reference Information Table (Plate III) in this report shows the number of the wire lots used in fabricating all fence and barbed wire samples.

Chain-Link Fence.—Chain-link fence is exposed at all locations except Ames, Manhattan, and College Station. At Pittsburgh rust is present on all except the two heaviest zinc-coated samples, and the corrosion-resistant steel samples. At Sandy Hook, Bridgeport, State College, Lafayette, and Ithaca rusting has occurred among the three lightest zinc-coated samples and the lead-coated samples. At Santa Cruz and Davis only the lead-coated samples show any rust.

Wire Strand.—Wire strand is exposed at all locations and shows pinhole rusting on the lead-coated strand at all locations. Only at Pittsburgh has rust appeared in the zinc-coated specimens. There, all but two samples (1.51 and 2.47 oz. per sq. ft.) are rusting.

Findings from Weight Loss Specimens:

At the start of the test 144 wires (16 alike of each of 9 wire lots) were exposed at Pittsburgh, at Sandy Hook, and at State College. The test procedure plan was to remove the specimens one fourth at a time from each location, taking the last quarter when the coating was nearly gone but before rusting started. All of the removals have been made from Pittsburgh and Sandy Hook, and half of the specimens have been removed from State College. The earlier removals from Pittsburgh, State College, and Sandy Hook were reported in the 1939 and 1941 *Proceedings*.[1] Table VII shows the results obtained from the fourth removals from Sandy Hook which were the only weight loss data assembled since the last published report. These data are in line with previously reported findings that:

1. There is a tendency for the lighter gages of wire to lose coating somewhat faster than the heavier gages.
2. There is an indication that there is slight decrease in the rate of coating loss with increasing periods of exposure.
3. The over-all average annual loss of coating in ounces per square foot of surface at Pittsburgh is 0.369; at Sandy Hook it is 0.117; and at State College it is 0.063.
4. There is not a consistent indication that any one of the three groups (A or B or C) loses coating at a more rapid rate than the other two, although the data might be interpreted to indicate that group C loses weight at the lowest rate.
5. The time required for first rust to appear on samples thus far rusted at Pittsburgh and at Sandy Hook checks reasonably well with the time that would be expected from the data of the weight loss specimens.

Findings from Specimens Removed for Tension Tests:

There were originally 840 tensile strength specimens exposed at each site, except Lafayette and College Station (833) and Davis and Santa Cruz (798). In the previously noted 1939 and 1941 *Proceedings*,[1] there were reported the results of tensile strength and elongation tests on 753 wires. Since then 284 additional wires have been removed, divided among the different test sites as shown in Table B. The detailed results of the tests on these 284 wires are shown in Table VI (Plate II).

TABLE B.—NUMBER OF TENSILE STRENGTH SPECIMENS REMOVED FOR TEST.

Exposure Site	Number of Wires Removed in 1941 and 1942				Wires Removed Prior to 1941	Total Removed to Date
	Bare Steel	Zinc Coated	Lead Coated	Corrosion Resistant		
Pittsburgh, Pa.	22	160	0	12	211	405
Sandy Hook, N. J.	15	24	0	0	109	148
Bridgeport, Conn.	23	21	0	...	86	130
State College, Pa.	7	0	0	0	45	52
All other locations	0	0	0	0	282	282
Total	67	205	0	12	733	1017

Pittsburgh is the most corrosive of the test sites, with Sandy Hook rating next, and Bridgeport next. Consequently more specimens have been removed from Pittsburgh than from any other location. Of the 133 uncoated mild steel wires exposed at Pittsburgh 107 (80 per cent) have been removed and of the 595 zinc-coated wires exposed at Pittsburgh 277 (46 per cent) have been removed. Of the 112 lead-coated, copper-coated, and corrosion-resistant wires exposed at Pittsburgh, 20 have been removed.

The one set of corrosion-resistant steel wires (high-chromium and nickle-chromium content wires) that were

TABLE C.—LOSS OF TENSILE STRENGTH SHOWN BY STEEL WIRES AFTER VARIOUS PERIODS OF EXPOSURE, PITTSBURGH, PA.

Wire Lot	Exposure Until First Rust, yr.	First Test			Second Test			Third Test			Fourth Test			Fifth Test			Sixth Test			Seventh Test		
		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr	Tensile Str Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss	
			per cent	per cent per year		per cent	per cent per year		per cent	per cent per year		per cent	per cent per year		per cent	per cent per year		per cent	per cent per year		per cent	per cent per year
ALL 500 SERIES (14½ GAGE) UNFABRICATED WIRES.																						
No. 501[a]	0	1.48	36.0	24	1.94	39.9	20	2.45	46.0	19	2.93	49.0	17	3.48	55.0	16	4.50	61.0	13	5.48	87.0	16
No. 506	0	1.48	32.7	22	1.94	35.5	18	2.45	37.5	15	2.93	45.2	15	3.48	48.1	14	4.50	57.8	13	5.48	82.7	15
No. 507	0	1.48	31.1	21	1.94	36.1	18	2.45	37.8	15	2.93	47.1	16	3.48	52.9	15	4.50	59.6	13	5.48	84.9	15
No. 510	1.20	0.28	0	0	0.74	10.0	13	1.25	14.0	11	1.73	25.0	15	2.28	31.0	14	3.72	46.0	12	4.75	65.0	14
No. 511	0.60	0.88	26.3	30	1.34	28.1	21	1.85	42.5	23	2.33	38.6	17	2.88	43.8	15	4.32	59.6	14	5.35	79.0	14
No. 512	0.50	0.98	26.1	27	1.44	27.7	19	1.95	30.8	16	2.43	41.5	17	2.98	44.6	15	4.42	49.2	11	5.45	83.0	15
No. 520	1.20	0.28	0	0	0.74	1.8	2	3.30	32.1	10	4.28	58.9	14	4.75	55.4	12						
No. 521	1.75	0	−3.7	0	0.20	−5.5	0	2.75	33.3	12	3.73	59.3	16	4.20	55.6	13						
No. 522	1.45	0	−2.0	0	0.50	5.0	10	3.05	40.0	13	4.03	65.0	16	4.50	64.0	14						
No. 530	1.20	0.28	−1.3	0	0.74	−4.5	0	3.72	50.0	13	4.28	72.6	17	4.75	62.9	13						
No. 531	1.40	0.08	8.0	0	0.54	15.0	28	3.52	50.0	14	4.08	63.0	15	4.55	66.0	15						
No. 540	1.85	0.10	−6.8	0	3.07	53.4	18	3.63	68.3	19	4.10	61.7	15									
No. 541	2.60	0	−1.5	0	2.32	32.9	14	2.88	65.7	23	3.35	57.2	17									
No. 542	1.65	0.30	0	0	3.27	41.2	13	3.83	57.8	15	4.30	59.8	14									
No. 543	1.85	0.10	−1.7	0	3.07	35.0	12	3.63	68.3	19	4.10	55.0	13									
No. 551	4.50	0.42	4.6	11																		
No. 552	4.90	0	−1.0	0																		
ALL 400 SERIES (12½ GAGE) UNFABRICATED WIRES.																						
No. 400[a]	0	1.48	25.0	17	1.94	23.0	12	2.45	25.0	10	2.93	32.3	11	3.48	32.3	9	4.92	43.8	9	5.95	54.2	9
No. 401[a]	0	1.48	28.9	20	1.94	25.5	13	2.45	31.1	13	2.93	38.0	13	3.48	41.2	12	4.92	50.0	10	5.95	65.6	10
No. 405	0	1.48	23.6	16	1.94	23.7	12	2.45	23.7	10	2.93	33.0	11	3.48	33.0	10	4.92	43.5	9	5.95	56.8	9
No. 406	0	1.48	21.3	14	1.94	21.3	11	2.45	22.3	9	2.92	27.7	9	3.48	28.7	8	4.92	37.2	7	5.95	44.7	7
No. 410	0.65	0.83	18.0	22	1.29	18.8	15	2.28	28.1	12	2.83	30.7	11	4.27	45.7	11	4.83	64.9	13	5.30	51.7	10
No. 411	0.85	0.63	7.3	12	1.09	9.7	9	2.08	16.3	8	2.63	26.8	10	4.07	31.8	8	4.63	41.5	9	5.10	45.5	9
No. 412	1.25	0.23	4.8	21	0.69	8.5	12	1.68	17.5	10	2.23	19.3	9	3.67	54.2	15	4.23	...	...	4.70	40.4	9
No. 421	1.60	0	4.3	0	0.34	3.2	10	2.90	25.5	9	3.88	40.4	10	4.35	28.6	7						
No. 422	1.45	0	8.0	0	0.50	5.0	10	3.05	23.0	8	4.03	40.0	10	4.50	34.0	8						
No. 423	1.25	0.23	17.5	...	0.70	17.5	25	3.25	25.0	8	4.23	41.2	10	4.70	40.0	9						
No. 430	2.60	0	−1.3	0	0	−4.5	0	2.32	15.4	7	2.88	35.3	12	3.35	38.5	11						
No. 431	1.60	0	6.8	0	0.34	11.4	33	3.32	23.9	7	3.88	41.5	11	4.35	39.8	9						
No. 432	1.60	0	−2.0	0	0.34	9.8	29	3.32	0	...	3.88	38.2	10	4.35	42.2	10						
No. 440	2.60	0	−6.8	0	2.32	31.4	14	2.88	39.8	14	3.35	41.6	12									
No. 441	1.90	0.55	−5.8	0	3.02	23.1	8	3.58	41.3	12	4.05	42.4	10									
No. 442	2.00	0.45	7.5	17	2.92	10.6	3	3.48	45.7	13	3.95	46.8	12									
No. 443	1.90	0.55	−1.3	0	3.02	22.0	7	3.58	38.0	11	4.05	41.4	10									
No. 444	2.70	0	0	0	2.22	21.1	10	2.78	37.7	14	3.25	43.8	13									
No. 495	1.90	0.55	−0.7	0	3.02	23.4	8	3.58	38.0	11	4.05	38.0	9									
No. 450	2.95	2.53	22.6																			
No. 451	4.50	0.98	21.6																			
ALL 300 SERIES (11 GAGE) UNFABRICATED WIRES.																						
No. 300[a]	0	1.48	20.1	14	1.94	18.2	9	2.45	19.6	8	2.93	24.3	8	3.48	23.6	7	5.95	40.5	7			
No. 302[a]	0	1.48	25.5	17	1.94	25.5	12	2.45	25.4	10	2.93	31.3	11	3.48	31.8	9	5.95	48.1	8			
No. 305	0	1.48	20.0	14	1.94	15.2	8	2.45	18.9	8	3.48	24.4	7	5.95	39.2	7						
No. 306	0	1.48	17.0	12	1.94	14.8	8	2.45	15.5	6	3.48	19.7	6	5.95	36.0	6						
No. 315	0.45	1.03	14.4	14	1.50	9.0	6	3.03	18.3	6	5.03	36.7	7									
No. 316	1.20	0.28	2.2	8	0.74	5.7	8	2.28	10.1	4	4.28	24.6	6									
No. 317	1.45	0.49	5.6	11	1.48	13.8	9	2.03	15.1	8	4.03	32.1	8									
No. 325	1.75	0	1.4	0	0.20	−2.1	0	3.17	9.6	3												
No. 326[a]	1.40	0.08	4.3	...	0.54	12.9	25	3.52	16.5	5												
No. 327	1.80	0	3.5	0	0.14	2.3	16	3.12	20.3	7												
No. 329	1.85	0	1.4	0	0.10	−4.2	0	3.07	16.7	5												

TABLE C.—(*Continued*)

Wire Lot	Exposure Until First Rust, yr.	First Test			Second Test			Third Test			Fourth Test			Fifth Test			Sixth Test			Seventh Test		
		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss		Exposure Since 1R, yr.	Tensile Str. Loss	
			per cent	per cent per year		per cent	per cent per year		per cent	per cent per year		per cent	per cent per year		per cent	per cent per year		per cent	per cent per year		per cent	per cent per year
All 300 Series (11 Gage) Unfabricated Wires.—(*Continued*)																						
No. 335	1.60	0.95	5.8	6	3.32	18.7	6	3.88	30.3	8												
No. 336	2.75	0	0	0	2.17	5.9	3	2.73	31.6	12												
No. 337	2.50	0	0	0	2.42	15.1	6	2.98	34.3	12												
No. 338	2.00	0.45	2.0	4	2.92	14.2	5	3.48	29.4	8												
No. 345	2.70	2.22	21.9	10	2.78	23.0	8															
No. 346	2.55	2.37	14.0	6	2.93	27.6	9															
No. 347	2.65	2.27	23.2	10	2.83	25.0	9															
No. 348	3.20	1.72	16.9	10	2.28	24.0	10															
No. 350	5.10	0.38	7.0	21																		
No. 351	2.90	2.58	13.9	5																		
No. 352	3.90	1.58	7.8	5																		
No. 397	...																					
All 200 Series (9 Gage) Unfabricated Wires.																						
No. 200[a]	0	1.48	20.5	14	1.94	19.2	10	2.93	25.8	9	3.48	26.8	8	5.95	35.9	6						
No. 202[a]	0	1.48	18.8	13	1.94	18.0	9	2.93	18.0	6	3.48	23.2	7	5.95	31.7	5						
No. 205	0	1.48	16.2	11	1.94	11.4	6	2.93	18.8	6	3.48	20.3	6	5.95	29.3	5						
No. 206	0	1.48	15.5	11	1.94	16.5	8	2.93	17.9	6	3.48	17.9	5	5.95	27.1	5						
No. 215	0.60	0.88	9.9	11	1.34	9.9	7	2.88	12.8	4	4.88	27.7	6	...	...	...						
No. 216	1.30	0.18	1.8	10	0.64	6.1	10	2.18	11.0	5	4.18	23.3	5	...	...	...						
No. 217	1.05	0.43	1.9	4	0.90	0	0	2.43	...	...	4.43	19.0	4	...	...	...						
No. 282	1.45	0.50	0.8	2	1.48	11.0	7	3.05	14.4	5	4.03	23.1	6	4.50	22.0	6						
No. 225	1.90	0	1.2	0	0.04	0.9	0	3.02	15.1	5												
No. 226	1.90	0	3.5	0	0.04	−1.7	0	3.02	16.5	5												
No. 227	2.50	0	3.0	0	0	4.3	0	2.42	20.0	8												
No. 228	1.60	0	0	0	0.34	−0.5	0	3.32	14.8	4												
No. 235	2.60	0.43	2.4	6	2.32	30.0	13	2.88	21.4	7												
No. 236	2.00	0.43	0	0	2.92	5.9	2	3.48	12.6	4												
No. 237	1.80	0.04	−2.8	0	3.12	15.9	5	3.68	19.5	5												
No. 238	2.50	0.04	−2.1	0	2.42	11.5	5	2.98	26.2	9												
No. 239	2.60	0	−0.9	0	2.32	11.6	5	2.88	20.1	7												
No. 245	2.40	2.52	14.6	6	3.08	18.8	6															
No. 246	2.90	2.02	8.8	4	2.58	15.6	6															
No. 247	2.90	2.02	12.3	6	2.58	23.5	9															
No. 248	2.10	2.82	14.1	5	3.38	19.3	6															
No. 296	2.50	2.32	10.8	5	2.98	15.3	5															
No. 801	3.50	1.42	22.5	16	1.98	30.2	15															
No. 250	3.90	1.58	7.2	4																		
No. 251	3.90	1.58	8.8	6																		
No. 252	4.50	0.98	13.5	13																		
No. 298	...																					
No. 299	...																					
All 100 Series (6 Gage) Unfabricated Wires.																						
No. 101[a]	0	1.48	13.4	9	1.94	13.4	7	2.93	14.5	5	3.48	18.1	5									
No. 105[a]	0	1.48	13.0	9	1.94	13.5	7	2.93	17.0	6	3.48	16.5	5									
No. 102	0	1.48	13.5	9	1.94	14.0	7	2.93	13.8	5	3.48	18.4	5									
No. 106	0	1.48	4.0	3	1.94	8.8	4	2.93	9.0	3	3.48	9.6	3									
No. 115	0.65	0.83	0.6	0	1.30	5.9	5	2.83	9.9	4	4.83	15.3	3									
No. 117	0.75	0.73	5.7	8	1.20	5.7	5	2.73	7.2	3	4.73	13.3	3									
No. 125	1.45	0	0	0	0.50	3.0	6	3.47	13.9	4												
No. 127[a]	1.70	0	−2.8	0	0.24	−0.8	0	3.22	18.7	6												
No. 135	2.60	0	3.1	0	2.32	12.4	5	2.88	16.5	6												
No. 138	2.00	0.45	2.0	4	2.92	9.7	3	3.48	14.3	4												
No. 139	1.90	0.55	1.6	3	3.02	11.2	4	3.58	16.1	4												
No. 148	2.75	2.17	7.1	3	2.73	11.6	3															
No. 149	2.75	2.17	6.3	3	2.73	13.1	5															
No. 800	3.90	1.02	6.9	7	1.58	9.4	6															
No. 151	3.90	1.58	6.6	4																		

[a] **Low copper content wires.**

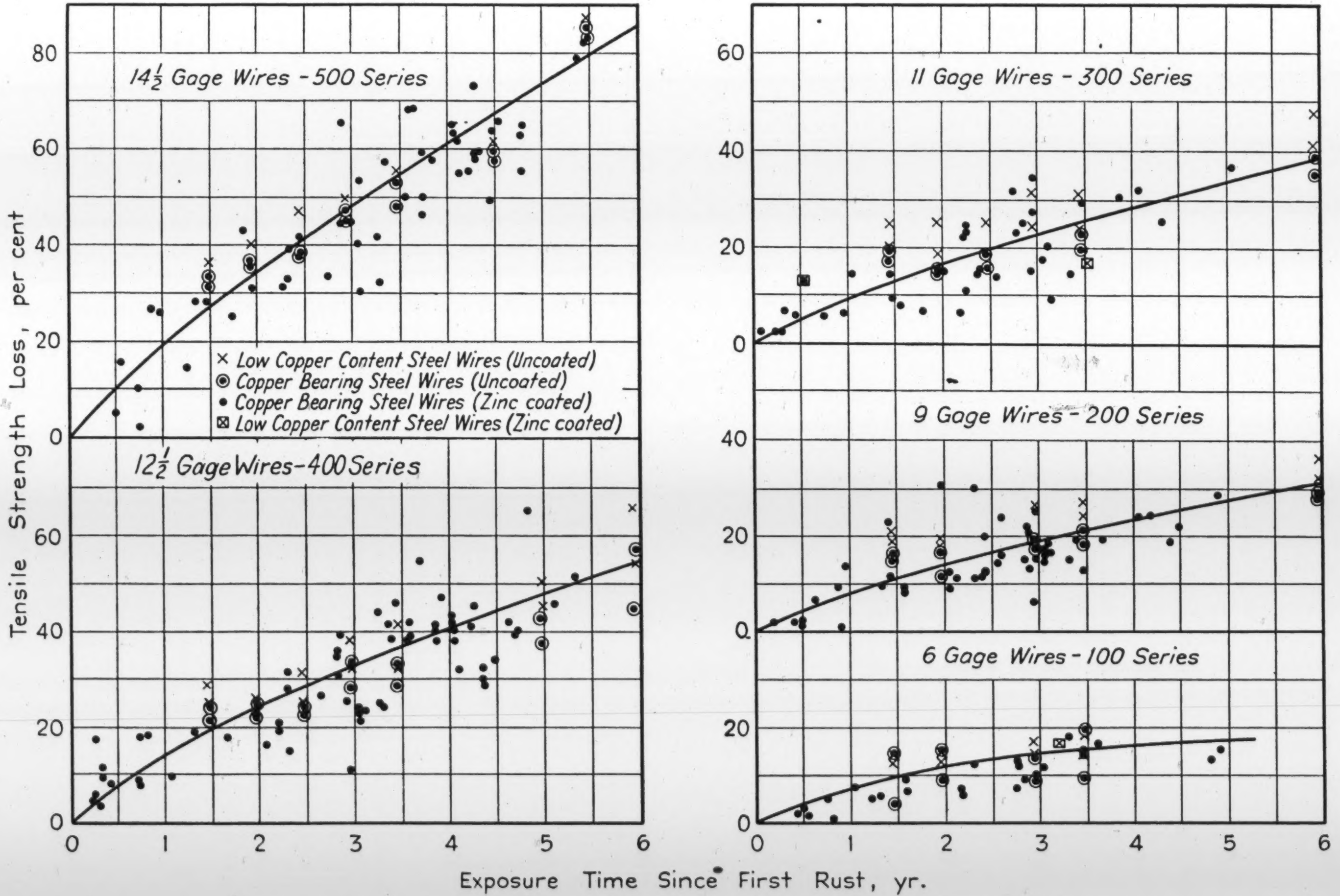

FIG. 3.—Tensile Strength Loss for Periods of Exposure Since First Rust, Pittsburgh, Pa.

removed after an exposure of about 5 yr. showed no loss in strength.

The set of copper-coated wires removed after about 5 yr. exposure showed no loss in strength.

No lead-coated wires were removed for tensile strength tests during 1941 and 1942 because tests on specimens previously removed (after exposures up to 3½ yr.) showed that no significant losses of strength had occurred. The specimens at that time showed considerable pinhole rusting. Apparently the pinhole rusting observed on these specimens, while undesirable from an appearance standpoint, does not materially affect the strength of the specimen. No very marked change in the appearance of the lead-coated samples has taken place since the last removals were made.

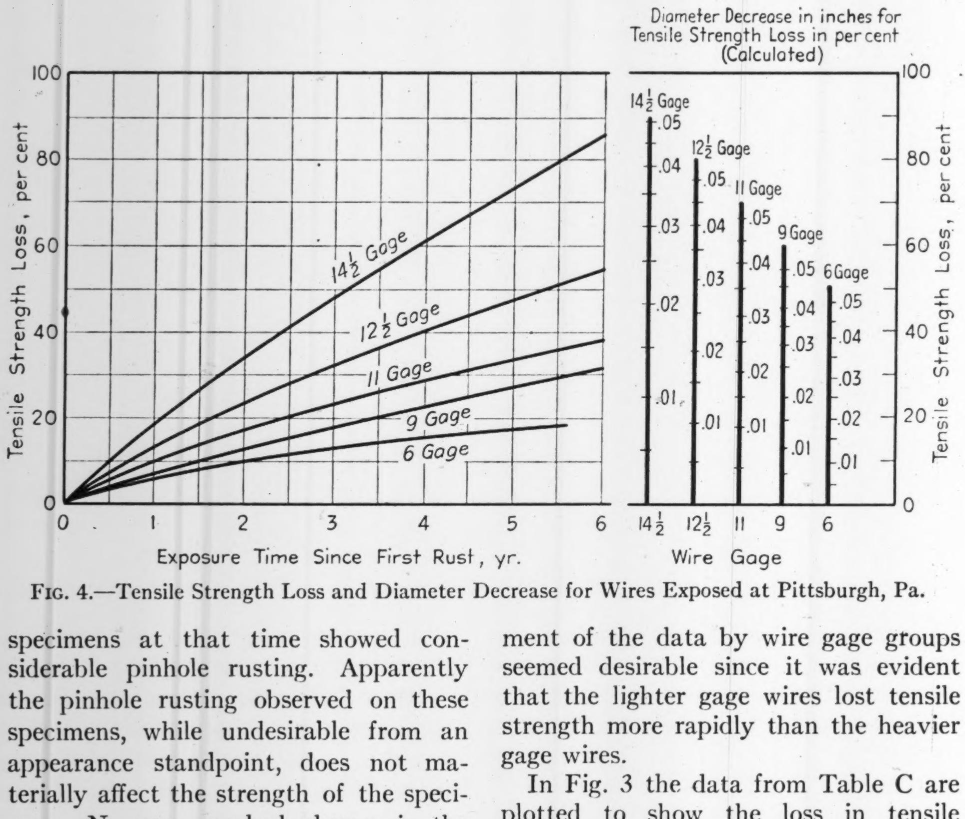

FIG. 4.—Tensile Strength Loss and Diameter Decrease for Wires Exposed at Pittsburgh, Pa.

In Table C are shown the detailed results of tensile strength tests on the 384 uncoated and zinc-coated mild steel specimens removed from the Pittsburgh test site. All the uncoated wires were assumed to have started to rust immediately on exposure. In the case of the zinc-coated specimens, the time of exposure, since first rust, was calculated by subtracting from the total time of exposure, the time required for first rust to appear as determined from the records of visual inspections. The table also shows, for each test specimen, the calculated tensile strength loss in per cent per year of exposure. Arrangement of the data by wire gage groups seemed desirable since it was evident that the lighter gage wires lost tensile strength more rapidly than the heavier gage wires.

In Fig. 3 the data from Table C are plotted to show the loss in tensile strength with different periods of exposure. All the test data is included except some few initial tests made before rusting occurred or so shortly after rusting started that the results were not significant. Through the plotted points a trend line for each wire gage was drawn by eye with the additional aid of short-cut statistical methods.

The 1941 wire test report indicated that the progressive loss of strength of zinc-coated wires after the zinc coatings were gone was about the same (or slightly slower) than the loss of strength experienced by uncoated wires. This is substantiated by the curves of Fig. 3 which show that the encircled points (copper-bearing uncoated steel wires) are in general not out of line with the nonencircled dots (copper-bearing zinc-coated steel wires).

Figure 4 shows these trend lines for all five wire gages under test and also shows five additional ordinate scales for diameter decreases corresponding to the tensile strength loss, so that a ready comparison can be made of both the depth of penetration of rust and percentage loss of tensile strength for the different gages of wire. The diameter decreases have been calculated from the formula:

$$\text{Diameter decrease} = D(1 - \sqrt{Sr})$$

where D = original wire diameter, and
Sr = percentage of initial strength remaining.

Corrosion at the other test site has not progressed sufficiently to make possible extensive comparisons of tensile strength loss. On the basis of present data it appears that at Sandy Hook the $14\frac{1}{2}$ gage wires lose tensile strength at about the rate for the $12\frac{1}{2}$ gage wires at Pittsburgh, and at Bridgeport the $14\frac{1}{2}$ gage wires lose tensile strength at about the rate for the 11 gage wires at Pittsburgh. Data from the remaining test sites are insufficient to permit comparisons.

The chairman of the Wire Test Inspection Committee wishes to express his appreciation to A. P. Jahn of the Bell Telephone Laboratories for his assistance in connection with the managerial work involved in the inspection of specimens, the task of analyzing data, and the preparation of this report.

CHEMICAL ANALYSIS OF COATINGS ON LEAD-COATED WIRES

The following is a report on recently completed chemical analyses made by the National Bureau of Standards of the coatings on wire samples taken from the same lots as the lead-coated wire undergoing exposure tests. These analyses were not made when the "referee" tests[3] were carried out at the Bureau (mostly in 1937 and 1938), but the wire which would have been used for analysis was saved.

The analyses reported below refer to the specimens shown in micrographs in Table I-0 of the 1939 Report of Committee A-5.[4] Examinations were made for tin and zinc contents which seem more significant in the characterization of lead coatings than analyses for cadmium, copper, and iron which were made in 1937 and 1938 to characterize the zinc coatings.

The coating weight determinations recently made do not check very well with those made at the time of the referee tests as shown in the following table:

	Wire Lot Number			
	B-040	B-041	B-042	B-043
Average diameter of wire, in.	0.188	0.144	0.117	0.08
Weight of coating, oz. per sq. ft.	1.60 1.40[a]	1.10 1.48[a]	1.73 1.68[a]	1.12 1.90[a]
Tin content, oz. per sq. ft.	0.004	0.002	0.003	0.005
Zinc content, oz. per sq. ft.	0.19	0.14	0.21	0.25

[a] Previously published coating weight determinations.[4]

Reference to the micrographs in the 1939 Report,[4] indicate that these lead-coated wires have a thin layer adjacent to the steel, differing in appearance from overlying lead. From these micrographs and the results of the analyses shown above it is reasonable to deduce that this underlying or bonding layer is zinc. Committee A-5 does not have under test at present any lead-coated wires which do not show this bonding layer. The committee does not wish to deduce or conclude that the performance of the specimens now on test is better, worse, or equivalent to specimens fabricated so that they are devoid of this bonding layer.

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[3] Report on Original Characteristics of Wire and Wire Products Involved in Field Exposure Tests, *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 101 (1939).

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 144 (1939).

Respectfully submitted on behalf of the Wire Test Inspection Committee,

C. D. HOCKER,
Chairman.

TABLE II.—REPORT OF INSPECTIONS OF BARBED WIRE SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. R = rust of base metal. PHR, PHY = rust or yellowed in pinholes, B, Br, Gn, respectively, signify corroded to a black, brown or green appearance, D = dark (dirt or soot) to the exclusion of a better observation. MG = intermediate M and G. GY predominately gray but showing indication of Y. RB = rust on barbs. SR = superficial rust. Figures (as 5, 20, 50, 100) are percentage's

Coating Group	Average Coating, oz. per sq. ft. of surface[a]	Wire[b]	State College, Pa.				Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
			3.51 yr.	3.87 yr.	4.42 yr.	5.10 yr.	5.05 yr.	5.99 yr.	5.28 yr.	6.07 yr.	4.95 yr.	5.94 yr.	4.93 yr.	6.01 yr.	4.83 yr.	5.86 yr.	4.82 yr.	5.82 yr.	4.89 yr.	5.86 yr.
0.20 to 0.30	0.30	No. C410...	3R	5R	30R	75R	20R	100R	G	20R	G	G	GYRB	90R	G	G	GY	10Y	G	G
	0.24	No. C411...	YRB	YRB	YRB	25R	25R	80R	GRB	10RRB	GRB	GRB	3RRB	20RRB	GRB	GRB	GRB	GRB	G	G
	0.29	No. C412...	YRB	GYRB	YRB	YRB	GRB	30R	G	G	G	G	GRB	5RRB	G	G	GY	12Y	G	G
0.35 to 0.45	0.37	No. C421...	Y	GY	Y	YRB	G	5R	G	G	G	G	G	GRB	G	G	GY	10Y	G	G
	0.47	No. C422...	YRB	GYRB	YRB	1RRB	GRB	25R	G	G	G	G	GRB	2RRB	G	G	GY	5Y	G	G
	0.31	No. C420...	Y	GY	YRB	2RRB	25R	80R	G	G	G	G	GRB	10RRB	G	G	GY	17Y	G	G
0.50 to 0.60	0.48	No. C430...	Y	GY	Y	Y	G	G	G	G	G	G	GRB	G	G	G	GY	10Y	G	G
	0.54	No. C431...	YRB	GYRB	YRB	YRB	GRB	GRB	G	G	G	G	G	1RRB	G	G	GY	25Y	G	G
	0.57	No. C432...	MG	MG	G	G	MG	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
0.70 to 0.85	0.72	No. C441...	MG	MG	G	G	MG	MG	G	G	MG	MG	M	MG	MG	MG	G	G	MG	MG
	0.78	No. C442...	M	MG	G	G	MG	MG	G	G	MG	MG	M	M	G	G	G	G	MG	MG
	0.74	No. C443...	MG	MG	G	GY	MG	MG	G	G	MG	MG	MG	MG	G	MG	G	G	MG	MG
0.80 to 1.00	0.97	No. C445...	MG	G	G	GY	G	G	G	G	MG	MG	MG	MG	G	G	G	G	MG	MG
	0.92	No. C446...	MG	G	G	GY	G	MG	G	G	MG	MG	MG	MG	MG	G	G	G	MG	MG
1.60 to 1.80	1.64	No. C450...	MG	G	G	G	G	G	G	G	MG	MG	MG	MG	G	MG	G	G	MG	MG
	1.50	No. C452...	M	G	G	G	MG	MG	G	G	MG	MG	M	M	G	G	G	G	G	G
	1.83	No. C451...	MG	G	G	G	MG	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
Heavy Coating	1.99	No. C494...	M	G	G	G	MG	M	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
	1.99	No. C494...	M	G	G	G	MG	M	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
	2.00	No. C447...	M	G	G	G	MG	MG	G	G	MG	MG	M	M	MG	G	G	G	MG	MG
Copper covered.......		No. C025...	Gn	GnRB	Gn	Gn	Gn	Gn	....	Br	Br	Br	Br	Br	BrRB	BrRB	Br	Br	BrRB	BrRB
(3.97 oz. per sq. ft)...		No. C025...	....	GnRB	....	....	....	30PHR	Br	....	....	....	....	Br	....	....	....	....	....	....
Lead coated..........		No. C045...	....	....	....	....	50R+Y	....	15PHY	15PHY	15PHY	15PHY	Br	3R	35SR	....	20R+Y	5R+Y	15SR	20SR
(1.83 oz. per sq. ft.)..		No. C045...	20R	....	....	....	....	....	10PHY	15PHY	....	....	5R	....	15SR	15SR	20R+Y	5R+Y	....	....

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[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabicated wire, fence or barbed wire of the specific wire lot number.

[b] These barbed wire numbers do not always correspond to the unfabricated wire numbers as seen below:

Barbed Wire	Unfabricated Wire	Barbed Wire	Unfabricated Wire
No. C410	No. 410	No. C441	No. 442
No. C411	....	No. C442	No. 441
No. C412	No. 412	No. C443	No. 443
No. C421	....	No. C445	....
No. C422	No. 421	No. C446	....
No. C420	No. 423	No. C447	....
No. C430	No. 431	No. C450	....
No. C431	No. 430	No. C452	No. 450
No. C432	No. 432	No. C451	No. 451
		No. C494	....

TABLE III.—REPORT OF INSPECTIONS OF FARM-FIELD FENCE SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. **R = rust of base metal.** PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively signify corroded to a black, brown, or green appearance, D = dark (dirt or soot) to the exclusion of a better observation. MG = intermediate between M and G. GY = predominately gray but showing indication of Y. SY or SR = superficial Y and superficial R, respectively. Asterisk (*) = speckled appearance. Figures (as 5, 20, 50, 100) are percentages. Where two values are shown (50R–80R) the first applies to the line wires and the second to the stay wires.

Coating Group	Average Coating, oz. per sq. ft. of surface[a]	Fence	State College, Pa.				Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
			3.43 yr.	3.87 yr.	4.42 yr.	5.10 yr.	5.05 yr.	5.99 yr.	5.28 yr.	6.07 yr.	4.95 yr.	5.94 yr.	4.93 yr.	6.01 yr.	4.83 yr.	5.86 yr.	4.82 yr.	5.82 yr.	4.89 yr.	5.86 yr.
0.20	0.27	No. 512....	Y	GY	Y	10R	50R–80R	100R	1R	100R–25R	GY	G	50R–50R	80R	G	G	5R	5R	G	G
to	0.30	No. 410....	Y	GY	5R	10R	50R–20R	100R	1R	25R–G	GY	G	5R–G	50R–10R	G	G	5R	5R	G	G
0.30	0.28	No. 411....	Y	GY	Y	Y	10R	G–70R	G	G	G	G	G	G–30R	G	G	GY	GY	G	G
Avg.	0.29	No. 510....	Y	GY	Y	1R	30R	80R	G	G	GY	G	G–10R	20R–90R	G	G	GY	GY	G	G
0.28	0.29	No. 412....	Y	GY	Y	5R	5R–30R	50R	G	G	G	G	G–10R	50R–60R	G	G	GY	5R	G	G
	0.24	No. 511....	Y	GY	Y	10R	40R–80R	G–100R	G	G	G	G	G–40R	60R–70R	G	G	GY	GY	G	G
0.25	0.25	No. 315....	GY	1R	G–20R	60R	45R–90R	100R	G–1R	50R–100R	G	G	2R–15R	70R	G	G	GY	7R	G	G
to	0.28	No. 215....	1R	1R	10R	10R	60R–80R	100R	10R–G	40R	GY	G	15R	90R	G	G	GY	5R	1R	G1R
0.35	0.38	No. 317....	GY	GY	Y	Y	G	10R	G	G	G	G	G	G–5R	G	G	GY	GY	G	G
Avg.	0.32	No. 316....	GY	GY	Y	Y	G	60R–30R	G	G	GY	G	G	10R	G	G	GY	GY	G	G
0.30	0.30	No. 216....	GY	GY	Y	Y	G	15R	G	G	GY	G	G	1R–20R	G	G	GY	GY	G	G
0.35	0.36	No. 520....	Y	GY	Y	Y	G	80R	G	G	G	G	G	1R	G	G	GY	GY	G	G
to	0.35	No. 422....	Y	GY	Y	Y	G–20R	G–80R	G	G	GY	G	G	1R–40R	G	G	GY	3R	G	G
0.45	0.31	No. 423....	Y	GY	Y	Y	G	G–30R	G	G	G	G	G	G–5R	G	G	GY	GY	G	G
Avg.	0.44	No. 522....	Y	GY	Y	Y	G	G	G	G	G	G	G	G	G	G	GY	GY	G	G
0.38	0.38	No. 521....	Y	GY	Y	1R	G–40R	G–100R	G	G	G	G	G	1R–5R	G	G	GY	GY	G	G
	0.47	No. 421....	Y	GY	Y	1R	G–20R	100R–G	G	G	G	G	G	G–10R	G	G	GY	GY	G	G
0.45	0.44	No. 325....	Y	GY	Y	Y	G	G	G	G	G	G	G	G	G	G	GY	GY	G	G
to	0.50	No. 228....	Y	GY	Y	Y	G	G	G	G	GY	G	G	G	G	G	GY	5R	G	G
0.55	0.51	No. 227....	Y	GY	Y	Y	G	G	G	G	G	G	G	G	G	G	GY	GY	G	G
Avg.	0.38	No. 326....	Y	GY	Y	Y	G	10R	G	G	G	G	G	G	G	G	GY	GY	G	G
0.46	0.47	No. 327....	Y	GY	Y	Y	G	G	G	G	GY	G	G	G	G	G	GY	GY	G	G
	0.45	No. 225....	GY	GY	Y	Y	G	G	G	G	G	G	G	G	G	G	GY	GY	G	G
0.50	0.61	No. 530....	MG	MG	G	G	MG	MG	G	G	MG	MG	M	1R	MG	MG	G	G	MG	MG
to	0.57	No. 432....	MG	MG	G	G	MG	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
0.60	0.60	No. 531....	MG	G	G	GY	G	G	G	G	MG	MG	MG	MG	MG	G	G	G	MG	MG
Avg.	0.48	No. 431....	Y	GY	Y	Y	G	G	G	G	G	G	G	G	G	G	GY	GY	G	G
0.54	0.54	No. 430....	Y	GY	Y	Y	G	G	G	G	G	G	G	G	G	G	GY	GY	G	G
	0.49	No. 532....	Y	GY	Y	Y	G	G	G	G	GY	G	G	G	G	G	GY	GY	G	G
	0.49	No. 433....	Y	GY	Y	Y	G	G	G	G	GY	G	G	G	G	G	GY	GY	G	G
0.60	0.69	No. 338....	MG	MG	G	G	MG	MG	G	G	MG	MG	M	G	MG	MG	G	G	MG	MG
to	0.68	No. 236....	MG	MG	G	G	MG	MG	G	G	MG	MG	M	G	MG	MG	G	G	MG	MG
0.70	0.61	No. 335....	MG	MG	GY	GY	G	G	G	G	MG	MG	MG	G	MG	G	G	G	MG	MG
Avg.	0.63	No. 237....	MG	MG	GY	GY	G	G	G	G	MG	MG	MG	G	MG	G	G	G	MG	MG
0.64	0.57	No. 337....	Y	GY	Y	Y	G	G	G	G	GY	G	G	G	G	G	GY	GY	G	G
	0.67	No. 235....	GY	GY	Y	Y	G	G	G	G	G	G	G	G	G	MG	GY	2R	G	G
	0.66	No. 336....	Y	GY	Y	Y	G	G	G	G	G	G	G	G	G	G	GY	GY	G	G
	0.60	No. 238....	GY	GY	GY	Y	G	G	G	G	G	G	G	G	G	G	GY	2R	G	G

0.70 to 0.85 Avg. 0.74	0.70	No. 540	MG	MG	G	G	MG	MG	G	G	MG	MG	M	M	MG	G	G	G	MG	MG
	0.72	No. 442	MG	MG	G	G	MG	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
	0.79	No. 543	MG	MG	G	GY	MG	MG	G	G	MG	MG	G	MG	MG	G	G	G	G	MG
	0.78	No. 441	MG	MG	G	G	MG	MG	G	G	MG	MG	M	MG	MG	MG	G	G	G	MG
	0.70	No. 542	MG	MG	G	GY	G	G	G	G	MG	MG	G	G	MG	G	G	G	MG	MG
	0.74	No. 443	MG	MG	G	GY	MG	MG	G	G	MG	MG	MG	MG	MG	G	G	G	MG	MG
	0.76	No. 541	MG	MG	G	GY	MG	MG	G	G	MG	MG	MG	MG	MG	G	G	G	MG	MG
	0.76	No. 440	MG	MG	G	G	MG	MG	G	G	MG	MG	M	M	MG	G	G	G	MG	MG
0.80 to 1.00 Avg. 0.90	1.05	No. 348	MG	MG	G	G	MG	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
	0.81	No. 248	MG	MG	G	G	MG	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
	0.88	No. 347	MG	MG	G	G	MG	MG	G	G	MG	MG	M	M	MG	G	G	G	G	MG
	0.95	No. 246	MG	MG	G	G	MG	MG	G	G	MG	MG	MG	MG	MG	G	G	G	MG	MG
	0.88	No. 346	MG	MG	G	GY	G	G	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
	0.81	No. 245	MG	MG	G	GY	G	G	G	G	MG	MG	MG	G	MG	G	G	G	MG	MG
	0.84	No. 345	GY	GY	GY	GY	G	G	G	G	G	G	G	G	G	G	G	G	G	G
	0.96	No. 247	MG	G	GY	GY	G	G	G	G	G	G	G	G	G	G	G	GY	G	G
1.60 to 1.80	1.75	No. 350	MG	MG	G	G	MG	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	M
	1.69	No. 252	MG	MG	G	G	MG	MG	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
	1.75	No. 352	MG	MG	G	G	MG	MG	G	G	MG	MG	M	M	MG	G	G	G	G	MG
Avg. 2.76	2.80	No. 397	MG	MG	G	G	MG	M	MG	MG	MG	MG	M	M	MG	G	G	G	MG	MG
	2.72	No. 298	MG	MG	G	G	MG	M	MG	MG	MG	MG	M	M	MG	MG	G	G	MG	MG
12–14% Cr, cold drawn		No. 001	M	M	M	MG	M	M	SY*	SY	MG*	MG*	M*	MG*	30SR	20SR	50SR	25SR	50SR	40SR
12–14% Cr, air quenched		No. 002	M	M	M	MG	M	M	MG	MG	MG	MG	M	M	15SR	G15SR	30SR	30SR	30SR	30SR
18% Cr, 8% Ni		No. 003	M	M	M	M	M	M	M	M	M	M	M	M	MG	MG	5SY*	5SY	MG	MG
Copper covered (7.42 oz. per sq. ft.)		No. 020	Gn	Gn	Gn	Gn	Gn	Gn	Br	Br	Br	Br	Br	Br	Br	Br	Br	Br	Br	Br
Lead coated (1.48 oz. per sq. ft.)		No. 040	30PHR	50R+Y	25R+Y	10R	20PHR	30R+Y	25PHY	40PHY	30PHY	30PHY	5R	10R	30SR	30R+Y	15PHSR	25PHSR	24SR	25SR*

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[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

TABLE IV.—REPORT OF INSPECTIONS OF CHAIN-LINK SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = Metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. R = rust of base metal PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance, D = dark (dirt or soot) to the exclusion of a better observation. MG = intermediate between M and G. GY = predominately gray but showing indication of Y. Figures (as 5, 20, 50, 100) are percentages.

All 200 series wires are 9 gage. Lead-coated wire, 9 gage. Corrosion-resistant steel, 14 gage.
All 100 series wires are 6 gage. Copper-covered wire 0.128d.

Zinc Coating, oz. per sq. ft. of surface, by stripping test	Fence	Pittsburgh, Pa.				Sandy Hook, N. J.				Bridgeport, Conn.			
		4.50 yr.	4.92 yr.	5.48 yr.	5.95 yr.	4.45 yr.	4.90 yr.	5.45 yr.	5.93 yr.	4.45 yr.	4.90 yr.	5.45 yr.	5.94 yr.
0.29	No. E280[a]	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
0.52	No. E281	100R	100R	100R	100R	50R	50R	80R	100R	20R	40R	40R	80R
0.37	No. E282	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
1.22	No. E185	25R	100R	100R	100R	GY	G	GY	GY	G	GY	GY	Y
1.13	No. E285	90R	100R	100R	100R	G	GY	GY	GY	G	G	GY	GY
2.92	No. E290	G	G	G	G	G	G	G	G	G	G	G	G
2.09	No. E291	GY	G	5R	40R	G	G	G	G	G	G	G	G
1.60	No. H280[a]	90R	90R	95R	90R	G	G	G	GY	G	G	GY	GY
1.83	No. H185	25R	90R	95R	95R	GY	G	G	GY	G	GY	GY	GY
1.60	No. H186	25R	95R	98R	98R	G	G	G	GY	G	G	GY	GY
1.61	No. H187	6R	90R	90R	90R	G	G	G	GY	G	G	GY	G
1.55	No. H188	60R	95R	98R	98R	G	G	G	GY	G	GY	GY	GY
1.36	No. H285	90R	95R	95R	85R	G	G	G	GY	G	G	GY	GY
1.97	No. H286	50R	95R	95R	90R	G	G	G	GY	G	G	GY	GY
1.46	No. H287	80R	95R	95R	95R	G	G	G	GY	G	GY	GY	GY
1.74	No. H288	35R	90R	98R	95R	G	G	G	GY	G	G	GY	GY
1.40	No. H289[b]	30R	100R	100R	100R	G	G	G	G	G	G	G	G
4.21	No. H290	GY	GY	GY	GY	G	G	G	G	G	G	G	G
Lead-coated L050 (1.75 oz. per sq. ft.)		5PHR	10PHR	5R+Y	15R	60R+Y	70R+Y	25R	25R	PHY	5R	1R	Y
Copper-covered K030 (8.25 oz. per sq. ft.)		....	....	....	....	Gn	Gn	Gn	Gn	Gn	Gn	Gn	Gn
Corrosion-resistant steel J011		M	M	M	MG	M	M	MG	M	M	M	M	M

Zinc Coating, oz. per sq. ft. of surface, by stripping test	Fence	State College, Pa.				Lafayette, Ind.		Ithaca, N. Y.		Santa Cruz, Calif.		Davis, Calif.	
		3.51 yr.	3.87 yr.	4.42 yr.	5.10 yr.	5.05 yr.	5.99 yr.	4.93 yr.	6.01 yr.	4.83 yr.	5.86 yr.	4.89 yr.	5.86 yr.
0.29	No. E280[a]	Y	GY	Y	15R	20R	70R	2R	90R	G	G	G	G
0.52	No. E281	GY	GY	GY	GY	G	G	G	G	G	G	G	G
0.37	No. E282	Y	GY	Y	Y	10R	60R	G	1R	G	G	G	G
1.22	No. E185	G	G	G	G	G	MG	MG	MG	G	MG	MG	MG
1.13	No. E285	MG	G	G	G	G	G	MG	MG	G	M	MG	MG
2.92	No. E290	G	G	G	G	MG	M	M	MG	G	MG	MG	MG
2.09	No. E291	G	G	G	G	MG	M	M	M	G	MG	MG	MG
1.60	No. H280[a]	G	G	G	G	MG	MG	MG	MG	MG	MG	MG	MG
1.83	No. H185	G	G	G	G	MG	MG	MG	MG	MG	MG	MG	MG
1.60	No. H186	G	G	G	G	MG	MG	MG	MG	G	MG	MG	MG
1.61	No. H187	G	G	G	G	MG	MG	MG	MG	G	MG	MG	MG
1.55	No. H188	MG	G	G	G	MG	MG	MG	MG	MG	MG	MG	MG
1.36	No. H285	MG	G	G	G	G	MG	M	MG	G	MG	MG	MG
1.97	No. H286	MG	G	G	G	G	MG	MG	MG	MG	MG	MG	MG
1.46	No. H287	MG	G	G	G	MG	MG	M	MG	MG	MG	MG	MG
1.74	No. H288	MG	G	G	G	MG	MG	M	MG	G	MG	MG	MG
1.40	No. H289[b]	MG	G	G	G	MG	MG	M	M	MG	MG	MG	MG
4.21	No. H290	MG	G	G	G	MG	M	M	M	MG	MG	MG	MG
Lead-coated L050 (1.75 oz. per sq. ft.)		50R+Y	55R+Y	50R+Y	10R	25PHY	20PHR	3R	1R	15SR	15SR	15SR	15SR
Copper-covered K030 (8.25 oz. per (sq. ft.)		Gn	Gn	Gn	Gn	Gn	Gn	Br	Br	BrRB	Br	Br	Br
Corrosion-resistant steel J011		M	M	M	M	M	M	M	M	M	MG	M	MG

[a] E numbers—coating applied before weaving. H numbers—coating applied after weaving.
[b] While this sample has an H number, its appearance indicates that it was galvanized before weaving.

TABLE V.—REPORT OF INSPECTIONS OF WIRE STRAND SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. R = rust of base metal. PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively signify corroded to a black, brown, or green appearance, D = dark (dirt or soot) to the exclusion of a better observation. MG = intermediate between M and G. GY = predominately gray but showing indication of Y. Figures (as 5, 20, 50, 100) are percentages.

Zinc Coating, oz. per sq. ft. of surface	Strand	Pittsburgh, Pa.				Sandy Hook, N. J.				Bridgeport, Conn.				State College, Pa.				Lafayette, Ind.	
		4.50 yr.	4.92 yr.	5.48 yr.	5.95 yr.	4.45 yr.	4.90 yr.	5.45 yr.	5.93 yr.	4.45 yr.	4.90 yr.	5.45 yr.	5.94 yr.	4.31 yr.	4.75 yr.	5.30 yr.	5.77 yr.	5.05 yr.	5.99 yr.
0.69	No. D345....	25R	50R	60R	70R	G	G	G	G	MG	G	G	G	MG	G	G	G	MG	MG
0.88	No. D346....	1R	10R	15R	35R	G	G	G	G	MG	G	G	G	MG	G	G	G	MG	MG
0.87	No. D347....	10R	65R	65R	95R	G	G	G	G	MG	G	G	G	MG	G	G	G	MG	MG
0.93	No. D348....	10R	60R	60R	20R	G	G	G	G	MG	G	G	G	MG	G	G	G	MG	MG
1.10	No. D360....	GY	20R	15R	30R	G	G	G	G	MG	G	G	G	MG	G	G	G	MG	MG
1.17	No. D361....	GY	5R	10R	50R	G	G	G	G	MG	G	G	G	MG	G	G	G	MG	MG
1.03	No. D362....	GY	15R	20R	50R	G	G	G	G	MG	G	G	GY	MG	G	G	G	MG	MG
1.51	No. D365....	GY	B	Y	GY	G	G	G	G	MG	G	G	G	MG	G	G	G	MG	MG
1.39	No. D366....	GY	5R	5R	15R	G	G	G	G	MG	G	G	G	MG	G	G	G	MG	MG
1.32	No. D367....	GY	1B	GY	5R	G	G	G	G	MG	G	GY	GY	MG	G	G	G	MG	MG
1.71	No. D371....	G	G	GY	2R	G	G	G	G	MG	G	G	G	MG	G	G	G	MG	MG
2.47	No. D375....	G	G	G	G	G	G	G	G	MG	G	G	G	MG	G	G	G	MG	MG
Lead Coating (1.96 oz. per sq. ft.) D045............		B	10PHR	D	10R	90R+Y	95R+Y	90R+Y	50R	50R+Y	50R+Y	15R+Y	55R	50R+Y	50R+Y	65R+Y	15R	60R+Y	65PHR

Zinc Coating, oz. per sq. ft. of surface	Strand	Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
		5.28 yr.	6.07 yr.	4.95 yr.	5.94 yr.	4.93 yr.	6.01 yr.	4.83 yr.	5.86 yr.	4.82 yr.	5.82 yr.	4.89 yr.	5.86 yr.
0.69	No. D345....	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
0.88	No. D346....	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
0.87	No. D347....	G	G	MG	MG	MG	MG	MG	G	G	G	MG	MG
0.93	No. D348....	G	G	MG	MG	M	M	G	G	G	G	MG	MG
1.10	No. D360....	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
1.17	No. D361....	G	G	MG	MG	MG	MG	MG	G	G	G	MG	MG
1.03	No. D362....	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
1.51	No. D365....	G	G	MG	MG	MG	MG	G	G	G	G	MG	MG
1.39	No. D366....	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
1.32	No. D367....	G	G	MG	MG	MG	MG	MG	MG	G	G	MG	MG
1.71	No. D371....	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
2.47	No. D375....	G	G	MG	MG	M	M	MG	MG	G	G	MG	MG
Lead Coating (1.96 oz. per sq. ft.) D045............		45PHY	50PHY	45PHY	5R	22R	50R	25SR	30SR	40R+Y	50R+Y	37SR	45SR

TABLE VII.—LOSS OF WEIGHTS OF ZINC-COATED WIRES.
(Tests on unfabricated wires weighed and measured before exposure.)
Losses of Weight in ounces per square foot.

Gage of Wire	Group A[a]			Group B[a]			Group C[a]			Averages of Like Gages
	Wire Lot	Rack Exposure		Wire Lot	Rack Exposure		Wire Lot	Rack Exposure		
		West Side	East Side		West Side	East Side		West Side	East Side	
SANDY HOOK EXPOSURES (4.45 YR.)										
6	No. 148	0.449 0.460	0.512 0.454							0.469
9				No. 247	0.553 0.509	0.554 0.537	No. 248	0.424 0.419	... ...	0.499
11	No. 346	0.520 0.523	0.544 0.520	No. 345	0.578 0.561	0.582 0.570	No. 348	0.433 0.431	0.449 0.442	0.513
14½	No. 542	0.558 0.523	0.549 0.554	No. 541	0.619 0.701	0.603 0.587	No. 540	0.465 0.465	0.468 0.469	0.547
	Average....	0.505	0.526	Average....	0.587	0.586	Average...	0.440	0.457	

[a] By reference to the published referee tests results, the wires listed under group A, (or B or C) will be found to be closely alike as regards base metal composition and coating characteristics.

REPORT OF COMMITTEE A-6
ON
MAGNETIC PROPERTIES

Two meetings of Committee A-6 on Magnetic Properties have been held during the past year, together with meetings of Subcommittees I, III and IV.

L. C. Hicks will succeed J. W. Woolridge as representative of the Allegheny Ludlum Steel Corp. on Committee A-6. Mr. Woolridge's recent death will be a great loss to the committee. He was a very active and valuable member for years.

With the development of the 28-cm. double lap joint specimen for a-c. testing, it soon became obvious that this same specimen could also be tested for d-c. magnetic properties without disturbing the sample in the test frame. A convenient method was to apply to the test frame extra coils suitable for this purpose and to connect them to the standard Fahy control box. If a set-up for testing ring samples is available, this may be used instead. Mr. S. L. Burgwin, who was responsible for the double lap joint sample, some time ago realized this possibility.

The Carnegie-Illinois Steel Corp. has recently made some tests to compare a-c. and d-c. permeability results as obtained by the methods mentioned above. These same samples have been checked on the Fahy Simplex permeameter and by the Westinghouse Electric and Manufacturing Co., using the double lap method.

A paper by J. P. Barton and G. W. Smith of the Carnegie-Illinois Steel Corp., entitled "Measurement of a-c. and d-c. Permeability on 28-cm. Test Specimens"[1] which describes these tests is to be presented at this annual meeting of the Society. This method may result in a considerable increase in speed of testing and simplification of test equipment, when both a-c. and d-c. results are required on the same samples. It is applicable, of course, only to laminated materials.

Subsequent to the 1942 annual meeting, the following three methods, which had been published as information as appendices to the 1942 annual report of Committee A-6, were revised, in the light of suggestions received, and presented to the Society through Committee E-10 on Standards for publication as tentative:

New Tentative Methods of Test for:

Permeability and Core Loss of Flat-Rolled Magnetic Materials Using 28-cm. Specimen (A 257 - 42 T),[2]

Incremental Permeability and Core Loss of Flat-Rolled Magnetic Materials at Low Alternating Inductions Using 28-cm. Specimen (A 258 - 42 T),[2] and

Permeability of Feebly Magnetic Materials (A 259 - 42 T).[2]

These new tentative methods were accepted[3] by Committee E-10 on August

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[1] See p. 493.

[2] 1942 Book of A.S.T.M. Standards, Part I, pp. 1091, 1097, 1103.

[3] In submitting these recommendations to Committee E-10 on Standards, Committee A-6 reported the following results of the letter ballot vote from a committee membership of 24: A 257 - 42 T, affirmative 12, negative 0, ballots marked "not voting" 2; A 258 - 42 T, affirmative 11, negative 0, ballots marked "not voting" 3; A 259 - 42 T, affirmative 13, negative 0, ballots marked "not voting" 1.

24, 1942, and assigned the designations indicated above.

I. Tentative Revisions of Standards

Standard Definitions of Terms, with Units and Symbols, Relating to Magnetic Testing (**A 127 – 41**).[4]—A. V. de Forest, a member of Committee A-6 and President of the Magnaflux Corp., has asked the committee to suggest and define another name for Magnaflux, to designate the use of magnetic powder or particles for the purpose of detecting structural defects in magnetic materials. The object of this request is to provide a descriptive name which is not subject to copyright limitations. The committee accordingly recommends that the following definitions and symbols be accepted for publication as tentative:

Magnetic Particle Method.—A method of detecting cracks and other structural defects in suitably magnetized materials, which employes the tendency of finely divided magnetic particles to congregate in regions of magnetic nonuniformity associated with the defects.

Note.—This method has been designated by the trade name "Magnaflux."

Apparent Power, P_a.—Apparent power is a term applied to the product of rms. voltage and rms. current in an a-c. circuit. For a-c. circuits generally, the unit is the "volt-ampere." For iron-cored circuits, specifically under condition of sinusoidal voltage, the unit is the "apparent watt."

Add the following symbols:

ν to represent magnetic reluctivity,
P_a to represent apparent power,
P_q to represent reactive power (vars),
N to represent the total number of turns in a winding, and
n to represent the number of turns per centimeter of length.

The above definition of apparent power should probably appear under "Core Loss, Apparent." Apparent watt values are, in general, much more valuable to the transformer designer than permeability values, since they enable him to calculate exciting currents directly, particularly since magnetizing current distortions and core loss effects are automatically included.

Standard Methods of Test for Magnetic Properties of Iron and Steel (**A 34 – 42**).[5]—The committee recommends for publication as tentative the following revision[6] of this standard:

Section 19 (*c*).—In the last formula and in Note 3 following this section, substitute the symbol P_q for the word "vars"; also in the legend under the formula change the second line to read as follows:

P_q = total reactive power in volt-amperes (vars),

Section 21 (*d*).—In the last formula and in Note 2 following this section, substitute the symbol P_q for the word "vars."

II. Editorial Change in Tentative Standard

Tentative Method of Test for Incremental Permeability and Core Loss of Flat-Rolled Magnetic Materials at Low Alternating Inductions Using 28-cm. Specimen (**A 258 – 42 T**).[7, 8]—The committee recommends the following editorial change in this method:

Section 6 (*b*).—Change the symbol for core loss from "P" to read "P_Δ" throughout this section.

Section 6 (*c*).—In conformity with the new symbol for reactive power, substitute the symbol P_q for the word "vars" in this section, and add "vars" after the formula.

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[4] 1942 Book of A.S.T.M. Standards, Part I, p. 665.

[5] 1942 Book of A.S.T.M Standards, Part I, p. 651.

[6] A further tentative revision of Standard Methods A 34 – 42, comprising a replacement of Sections 13 to 21 by a reference to the Tentative Method of Test for Normal and Incremental Permeability and Core Loss of Flat-Rolled Magnetic Materials at Low Alternating Inductions Using 28-cm. Specimen (A 258 – 43 T), was accepted by Committee E-10 on Standards and supersedes this tentative revision, see Editorial Note, p. 99.

[7] 1942 Book of A.S.T.M. Standards, Part I, p. 1097.

[8] See Editorial Note, p. 99.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 24 members; 17 members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. Tentative Revisions of Standards			
Def. of Terms, with Units and Symbols, Relating to Magnetic Testing (A 127 – 41)	16	1	0
Test for Magnetic Properties of Iron and Steel (A 34 – 42)	17	0	0
II. Editorial Change in Tentative Standard			
Test for Incremental Permeability and Core Loss of Flat-Rolled Magnetic Materials at Low Alternating Inductions Using 28-cm. Specimen (A 258 – 42 T)	17	0	0

Activities of Subcommittees

Subcommittee I on Nomenclature and Definitions (P. H. Dike, chairman).—In addition to the new symbols and definitions being recommended for publication as tentative, further changes are contemplated during the coming year. With the publication of the American Standard Definitions of Electrical Terms, developed by the American Institute of Electrical Engineers and approved by the ASA, it became evident that there are certain important differences between these definitions and those of the A.S. T.M. Subcommittee I will make an attempt this year to bring the A.S.T.M. definitions into closer agreement with the American Standard where it seems feasible to do so. In some instances the A.I.E.E. definitions are too complicated and difficult to understand to be of much service to the average magnetician, and simpler definitions will be developed or retained by the Society. On the other hand, many of the A.I.E.E. definitions can be adopted verbatim for A.S.T.M. use. In a few instances, where errors exist, an attempt will be made to bring about modification of the A.I.E.E. definitions in the next issue of this valuable publication. Some improvement in A.I.E.E. magnetic symbols also seems possible.

Subcommittee III on Direct Current Test Methods (B. M. Smith, chairman).—As mentioned in the report last year, a third method of testing feebly magnetic materials, developed by the Leeds & Northrup Co., is under consideration for approval as tentative. It is expected that this method will shortly be ready for submittal to the Society.[8] It is in commercial use and for certain applications offers advantages over those included in the Tentative Methods of Test for Permeability of Feebly Magnetic Materials (A 259 – 42 T).

Based on the information contained in the paper by J. P. Barton and G. W Smith, "The Measurement of a-c. and d-c. Permeability on 28-cm. Test Specimens,"[1] this subcommittee will prepare a standard specification for this type of direct current testing.

Subcommittee IV on Alternating Current Test Methods (J. P. Barton, chairman).—This committee is very active as usual, and two new major projects are under way.

When Tentative Method A 258 – 42 T, dealing with incremental magnetic properties, was prepared, it was thought that this test might be modified to include the procedure dealing with alternating current tests at low inductions which now appear in the Standard Methods of Test for Magnetic Properties of Iron and Steel (A 34 – 42).[8] In fact, if the polarizing current is reduced to zero such a test results. The bridge circuits of the newer test methods have certain decided advantages over bridge circuits of the older method. This subcommittee will soon combine these two methods which will result in the elimination of certain duplication and provide a better low-induction test method.

Many important applications of power supplies, with frequencies in excess of 60 cycles per second, are now being made, particularly up to 10,000 cycles. Two conspicuous examples are alternating-current systems for aircraft and frequencies of several thousand cycles for a wide variety of induction heating of ferrous materials. This has resulted in a demand for high frequency core loss data on electrical sheet and no standard method of test is available. Subcommittee IV, therefore, is undertaking the preparation of a procedure for measuring core loss at frequencies up to 10,000 cycles per second. This will probably be a wattmeter method with special precautions for reducing and determining the instrument errors due to these high frequencies. Another difficulty will be the very considerable heating of the samples at the higher frequencies and inductions. As a preliminary step in the preparation of this high frequency method, a small number of sheet samples will be circulated between a few laboratories equipped to make such tests, in order to determine whether good agreement can readily be obtained. If appreciable discrepancies develop, this will indicate the necessity for taking greater precautions than are now customary.

With magnetic core temperatures now often exceeding 100 C., the question was raised concerning the effect on aging, since our standard test is at 100 C. It was agreed that no adequate data are available to serve as a basis for a higher temperature specification. There is a little evidence to indicate that the maximum aging occurs below 150 C. for commercial silicon steel. It is urged that those having suitable facilities undertake to obtain aging data on commercial electrical sheet at temperatures above 100 C.

This report has been submitted to letter ballot of the committee which consists of 24 members; 17 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

THOMAS SPOONER,
Chairman.

R. L. SANFORD,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-6 presented to the Society through Committee E-10 on Standards the following recommendations:

Revision of Tentative Methods of:

Test for Permeability and Core Loss of Flat-Rolled Magnetic Materials Using 28-cm. Specimen (A 257 – 42 T),

Test for Incremental Permeability and Core Loss of Flat-Rolled Magnetic Materials at Low Alternating Inductions Using 28-cm. Specimen (A 258 – 42 T),

Test for Permeability of Feebly Magnetic Materials (A 259 – 42 T), and

Tentative Revision of Standard Methods of:

Test for Magnetic Properties of Iron and Steel (A 34 – 42).

These recommendations were accepted by Committee E-10 on August 30, 1943, and the revised tentative standards and the tentative revision appear in the 1943 Supplement to the Book of A.S.T.M. Standards, Part I, pp. 209, 215, 223 and 349.

REPORT OF COMMITTEE A-7

ON

MALLEABLE IRON CASTINGS

Committee A-7 on Malleable Iron Castings has held two meetings during the past year: one on June 24, 1942, during the Annual Meeting of the Society in Atlantic City, N. J., and the other on October 16, 1942, in Cleveland, Ohio.

At the request of the War Production Board, due to impending shortages of copper, a special committee was appointed to develop specifications for malleable iron flanges, pipe fittings, and valve parts. The committee met promptly, ironed out all difficulties, and submitted to Committee E-10 on Standards proposed Emergency Specifications for Malleable Iron Flanges, Pipe Fittings, and Valve Parts. This recommendation was accepted by Committee E-10 on October 6, 1942, and the emergency standard appears in the 1942 Book of A.S.T.M. Standards, Part I, bearing the A.S.T.M. serial designation ES - 20. It is felt that these specifications have been of considerable value in connection with the use of malleable castings in place of copper-bearing materials.

At the Cleveland meeting, Subcommittee IV on Pearlitic and Alloy Malleable Iron (C. F. Lauenstein, chairman) presented proposed tentative specifications for pearlitic malleable castings. This matter still proved to be controversial and it was impossible for the main committee to agree on the specifications. They were therefore referred back to the subcommittee. Subcommittee IV has been gathering information on this subject, but at the present time no specifications have met with the approval of the main committee. In view of the difficulty of members getting to the annual meeting this year, Committee A-7 is not planning to hold a meeting at that time, but the revised specifications for pearlitic malleable castings will be submitted by letter to the members of the committee for comment.

This report has been submitted to letter ballot of the committee which consists of 43 members, 28 members returned their ballots, of whom 26 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

E. K. Smith,
Chairman.

H. A. Schwartz,
Secretary.

REPORT OF COMMITTEE A-10

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL, AND RELATED ALLOYS

Committee A-10 on Iron-Chromium, Iron-Chromium Nickel, and Related Alloys held two meetings during the year: in Atlantic City, N. J., on June 24, 1942, and in Buffalo, N. Y., on March 4, 1943. The committee also met during the Annual Meeting of the Society in Pittsburgh, Pa., on June 28, 1943, prior to the presentation of this report.

Several changes in committee personnel occurred during the year. The present membership is 80 of whom 36 are classified as producers, 32 as consumers, and 12 as general interest members.

J. J. B. Rutherford has succeeded Newell Hamilton as chairman of Subcommittee XI on Specifications for Tubular Products.

At the request of the Administrative Committee, N.E.S.S., work has been undertaken on the formulation of specifications for stainless steel tubing to be prepared under the joint sponsorship of Committee A-1 on Steel and Committee A-10. Members of Subcommittee IX on Steel Tubing and Pipe of Committee A-1 are serving jointly with members of Subcommittee XI on Specifications for Tubular Products of Committee A-10, with J. J. B. Rutherford as chairman of the joint group.

D. B. Rossheim is serving as chairman of a subgroup of Subcommittee IX on Specifications for Flat Products delegated to prepare specifications for composite corrosion-resisting clad plate sheet and strip. This is a well balanced special committee comprising representatives of four producers, five fabricators, and four consumers.

I. New Tentative Standard

The committee recommends that the Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels[1] be accepted for publication as tentative.

II. Revision of Standards, Immediate Adoption

The committee recommends for immediate adoption revisions in four specifications, as indicated below, and accordingly asks for the necessary nine-tenths vote in order that these modifications may be referred to letter ballot of the Society:

Standard Specifications for Corrosion-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip (**A 167 - 42**):[2]

Table I.—Make the changes in the requirements as to chemical composition of the grades indicated in the accompanying Table I, and omit the requirements for grades 9 and 12.

Table II.—Omit the requirements as to physical properties for grades 9 and 12, and change footnote *a* from its present form: namely,

> [a] Yield strength shall be defined as the stress causing 0.5 per cent extension under load.

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[1] This recommended practice was accepted as tentative by the Society and appears in the 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 205.

[2] 1942 Book of A.S.T.M. Standards, Part I, p. 449.

to read as follows:

[a] See Section 7 (*b*).

TABLE I.

Grade	Type Number	Element	Change Present Requirements From	To Read
	301	Silicon, per cent....	0.75 max.	1.00 max.
	302	Silicon, per cent....	0.75 max.	1.00 max.
3	304	Silicon, per cent....	0.75 max.	1.00 max.
4	308	Silicon, per cent....	0.75 max.	1.00 max.
		Chromium, per cent.	19.00 to 22.00	19.00 to 21.00
5	321	Silicon, per cent....	0.75 max.	1.00 max.
		Nickel, per cent....	8.00 min.	8.00 to 11.00
6	347	Silicon, per cent....	0.75 max.	1.00 max.
		Nickel, per cent....	8.00 min.	9.00 to 12.00
7	302B	Silicon, per cent....	2.00 min.	2.00 to 3.00
11	316	Silicon, per cent....	0.75 max.	1.00 max.
		Molybdenum, per cent..............	2.00 to 3.00	1.75 to 2.50

Section 7 (*b*).—Change from its present form: namely,

> (*b*) The yield strength shall be determined by the dividers method.

to read as follows:

> (*b*) The yield strength shall be determined by the offset method as described in the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8). The limiting permanent offset shall be 0.2 per cent of the gage length of the specimen. An alternative method of determining yield strength may be used based on a total extension under load of 0.0063 in. in a 2-in. gage length.

Standard Specifications for Corrosion-Resisting Chromium Steel Plate Sheet, and Strip (**A 176 – 42**):[3]

Table I.—Make the following changes in the requirements as to chemical composition:

Grade	Type Number	Element	Change Present Requirements From	To Read
1	403	Phosphorus, per cent..	0.030 max.	0.040 max.
		Sulfur, per cent........	0.030 max.	0.040 max.
		Silicon, per cent.......	0.75 max.	1.00 max.
		Manganese, per cent...	0.60 max.	1.00 max.
2	410	Phosphorus, per cent...	0.030 max.	0.040 max.
		Sulfur, per cent........	0.030 max.	0.040 max.
		Silicon, per cent.......	0.75 max.	1.00 max.
		Manganese, per cent...	0.60 max.	1.00 max.
		Chromium, per cent...	10.00 to 14.00	11.50 to 13.50
4	430	Phosphorus, per cent...	0.030 max.	0.040 max.
		Sulfur, per cent........	0.030 max.	0.040 max.
5	442	Phosphorus, per cent..	0.030 max.	0.040 max.
		Sulfur, per cent........	0.030 max.	0.040 max.
6	446	Phosphorus, per cent...	0.030 max.	0.040 max.
		Sulfur, per cent........	0.030 max.	0.040 max.
		Chromium, per cent...	23.00 to 30.00	23.00 to 27.00

Standard Specifications for High-Strength Corrosion-Resisting Chromium-Nickel Steel Sheet and Strip (**A 177 – 39**):[4]

Section 4.—Change the chemical requirements for silicon from "0.75" to read "1.00" max., per cent.

Section 8 (*b*) *and* (*c*).—Replace these paragraphs which read as follows:

> (*b*) The yield point shall be determined by the dividers method.
>
> (*c*) In making tension tests reference should be made to the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.

by the following:

> (*b*) The yield strength shall be determined by the offset method as described in the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8). The limiting permanent offset shall be 0.2 per cent of the gage length of the specimen. An alternative method of determining yield strength may be used, based on the following total extensions under load:

Yield trength, min., psi.	Total Extension Under Load in 2-in. Gage Length, in.
75 000............	0.0098
110 000............	0.0125
135 000............	0.0144
140 000............	0.0148

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[3] 1942 Book of A.S.T.M. Standards, Part I, p. 460.

[4] *Ibid.*, p. 456.

Standard Specifications for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (**A 240 – 42**):[5]

Table I.—Make the changes in the requirements as to chemical composition shown in the accompanyng Table II.

TABLE II.

Grade	Type Number	Element	Change Present Requirements From	To Read
S	304	Silicon, per cent	0.75 max.	1.00 max.
		Chromium, per cent	18.00 min.	18.00 to 20.00
		Nickel, per cent	8.00 min.	8.00 to 10.00
M	316	Carbon, per cent	0.07 max.	0.10 max.
		Silicon, per cent	0.75 max.	1.00 max.
		Chromium, per cent	17.00 min.	16.00 to 18.00
		Nickel, per cent	10.00 min.	10.00 to 14.00
		Molybdenum, per cent.	2.00 min.	1.75 to 2.50
C	347	Silicon, per cent	0.75 max.	1.00 max.
		Chromium, per cent	17.00 min.	17.00 to 19.00
		Nickel, per cent	9.50 min.	9.00 to 12.00
T	321	Silicon, per cent	0.75 max.	1.00 max.
		Chromium, per cent	17.00 min.	17.00 to 19.00
		Nickel, per cent	9.00 min.	8.00 to 11.00

Omit the requirements for grade A, type 430 and grade B, type 446, and substitute the chemical requirements for grade A, type 410 and grade B, type 430 as given in the accompanying Table III.

TABLE III.

Grade	Type	Carbon, max., per cent	Manganese, max., per cent	Phosphorus, max., per cent	Sulfur, max., per cent	Silicon, max., per cent	Chromium per cent	Nickel, max., per cent
A	410	0.15	1.00	0.04	0.04	1.00	11.50 to 13.50	0.60
B	430	0.12	1.00	0.04	0.04	1.00	14.00 to 16.00	0.60

Table II.—Omit the requirements for grade A, type 430 and grade B, type 446, and substitute the physical test requirements for grade A, type 410 and grade B, type 430 as given in the following table:

Grade	Type	Tensile Strength, min., psi.	Yield Strength min., psi.	Elongation in 2 in., min., per cent	Hardness		Cold Bend, deg.
					Brinell Number, max.	Rockwell Number, max.	
A	410	70 000	35 000	25.0	202	B88	180
B	430	70 000	35 000	22.0	202	B88	180

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 80 members with the results shown in Table IV.

TABLE IV.—ANALYSIS OF LETTER BALLOT VOTE

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Rec. Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels	52	2	2
II. REVISION OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Corrosion-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip (A 167 – 42)	47	0	6
Spec. for Corrosion-Resisting Chromium Steel Plate, Sheet, and Strip (A 176 – 42)	47	0	6
Spec. for High-Strength Corrosion-Resisting Chromium-Nickel Steel Sheet and Strip (A 177 – 39)	47	0	6
Spec. for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (A 240 – 42)	46	1	6

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Classification of Data (E. F. Cone, chairman; Russell Franks, secretary).—Last year the data on wrought materials collected by this subcommittee were compiled and published in the form of "Tables of Data on Chemical Compositions, Physical and Mechanical Properties of Wrought Corrosion-Resisting and Heat-Resisting Chromium and Chromium-Nickel Steels."[6] The subcommittee is now engaged in collecting similar information for alloys in cast form. The Alloy Castings Institute, which represents the producers of high-alloy chromium and chromium-nickel steel castings, is cooperating with the Society and will assist in obtaining the data from its members. A form of data sheet for collecting the data has been agreed upon and will be distributed in the near future. It is estimated that approximately six months will be required to get the data following which it will be correlated and published as a set of companion tables to those published on wrought alloys. Some twenty-five alloys may be included although the list may be limited to include only those which have considerable commercial demand in heat- or corrosion-resisting services.

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[5] 1942 Book of A.S.T.M. Standards, Part I, p. 467.

Subcommittee IV on Methods of Corrosion Testing (F. L. LaQue, chairman). —This subcommittee has prepared a Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels which is recommended for publication as tentative. A standard test procedure was requested by the Welding Research Committee of the American Welding Society for use in connection with the testing of welds. In addition, several consumers have used a boiling nitric acid acceptance test for stainless steels and there therefore appeared to be a need for a standardized procedure for such testing.

The subcommittee is starting the preparation of a method of immersion testing similar to that being developed by Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys. The B-3 method[7] will either be adapted to Committee A-10's needs or a suitable form of test will be developed.

C. C. Snyder is continuing to cooperate with Committee B-3 as the representative of Committee A-10 in connection with the atmospheric-galvanic couple test being made by Subcommittee VIII on Galvanic and Electrolytic Corrosion, of Committee B-3. Subcommittee VIII of Committee B-3 is proposing to add some combinations of type 430 stainless steel and 24 ST aluminum alloy to these tests. Recommendation has been made by Committee A-10 to Committee B-3 to expose new couples of carbon steel and stainless steel inasmuch as the original couples of carbon steel contained only 0.018 per cent copper and were corroded at an abnormally high rate in marine and industrial atmospheres. It was suggested that the new couples of carbon steel contain 0.05 to 0.10 per cent copper as representing that likely to be obtained commercially.

The subcommittee is to outline a program for atmospheric exposure tests of stainless steels and was instructed by the main committee to give consideration to one industrial and two marine atmospheric test sites. The tests are expected to establish durability by visual examination and also by change in mechanical properties; in the latter connection consideration is being given to the measurement of endurance limit. The program which will be submitted to the committee when prepared may involve not only plain sections or sheets but also spot-welded, stressed or cold-worked forms such as may be used in architectural or engineering structures or transportation equipment.

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[6] Issued December, 1942.

[7] Tentative Method of Total Immersion Testing of Non-Ferrous Metals (B 185 - 43 T), see 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 332.

Reinspection of existing architectural installations will also be made by Mr. Snyder's subgroup with examination of stainless steel trains and buildings not previously examined.

Subcommittee V on Mechanical Tests (T. F. Olt, chairman).—During the past year, Subcommittee V has held two meetings at the first of which a cooperative test program was organized to obtain information on mechanical testing of sheet samples of several of the various stainless steel grades. This round-robin test program was conducted by five companies on seven different grades of stainless steel, and the results were analyzed and discussed at the Spring Meeting held on March 4, 1943, in Buffalo, N. Y. As a result of these tests, sufficient data were secured to indicate that specifying of the yield point determined by the dividers method was likely to yield results with such a high degree of inaccuracy, that it was advisable to change the specifications to require the yield strength to be determined by the offset method. A motion was passed that Subcommittee V recommend to Subcommittee IX that the yield point provisions of Specifications A 167 and A 177 be changed to yield strength established as 0.2 per cent offset, allowing as an alternate the extension under load method as now employed in the Army-Navy Specification for Corrosion-Resisting Steel (18 Cr – 8 Ni) Plate, Sheet, and Strip (AN-QQS-772). The allowable extensions under load for the various yield strengths when using this alternate method are as follows:

Yield Strength, min., psi.	Total Extension Under Load in 2-in. Gage Length, in.
30 000	0.0063
75 000	0.0098
110 000	0.0125
135 000	0.0144
140 000	0.0148

Additional round-robin investigations are now under way to determine the effect of variations in rate of strain in conducting the tension test, and also to determine the effect of variations in sample preparation. It is hoped to have these data available by the next spring meeting.

Subcommittee VI on Metallography (Russell Franks, chairman).—Last year, this subcommittee completed its second report on "Metallographic Examination of 18 per cent Chromium, 8 per cent Nickel Steel"[8] to the effect that it is impossible to determine whether a steel of the 18 per cent chromium, 8 per cent nickel type is subject to intergranular corrosion simply by etching polished surfaces of the steel and examining them under the microscope. It is anticipated that work of this nature will be continued with the object of finding an etching reagent that can be more intelligently used to establish whether the steel is subject to this defect. While it is doubtful whether such a test can be easily developed, it can readily be appreciated that if a procedure of this kind could be developed it would greatly lessen the time now required to determine the susceptibility of a given 18 per cent chromium, 8 per cent nickel steel to intergranular attack. The future program of this subcommittee is being given consideration.

Subcommittee VII on Welding (L. C. Bibber, chairman).—The function of Subcommittee VII is to keep informed on the activities of the various committees dealing with the welding of stainless steels and to apprise Committee A-10 thereof.

The Engineering Foundation Welding Research Council Committee on High-Alloy Steels is continuing its investigations on the various phases of the welding of the stainless steels.

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[8] *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 134 (1942).

The American Welding Society's Committee on High Alloy Engineering Structures has been disbanded.

Subcommittee VIII on Specifications for Wrought Products (J. K. Findley, chairman).—This subcommittee has in preparation specifications for hot-rolled and cold-rolled corrosion-resisting steel bars which will include ten grades of chromium and chromium-nickel alloy steels. These will be designated by the usual A.I.S.I. grade numbers. The specifications will not include any corrosion test requirements. After the specifications for bar stock have been issued, attention will be given to the formulation of specifications for corrosion-resisting wire and for forging billets.

Subcommittee IX on Specifications for Flat Products (C. C. Snyder, chairman).—Subcommittee IX recommends for immediate adoption changes in the chemical composition requirements of Specifications A 167, A 176, A 177, and A 240, these modifications being required due to the use of greater amounts of stainless steel scrap in melting in order to conserve critical alloying materials. Based on information supplied by Subcommittee V, a recommendation is also made to change the method of yield point determination in Specifications A 167 and A 177 to yield strength of limiting permanent offset of 0.2 per cent of the gage length of the specimen using as an alternative method total extension under load. The detailed changes in these specifications are given earlier in this report.

During the year, a subgroup under the chairmanship of D. B. Rossheim was formed to develop specifications on composite corrosion-resisting clad materials. The personnel of this subgroup is as follows:

Producers:
F. G. Flocke, The International Nickel Co.
W. R. Grunow, Allegheny Ludlum Steel Corp.
S. L. Ingersoll, Ingersoll Steel and Disc Division.
W. G. Theisinger, Lukens Steel Co.
L. W. Townsend, Composite Steel Division.
Fabricators:
E. C. Chapman, Combustion Engineering Co.
L. H. Johnson, Struthers-Wells
A. R. McAllister, The Babcock & Wilcox Co.
D. B. Rossheim, The M. W. Kellogg Co.
C. W. Wheatley, The A. O. Smith Corp.
Consumers:
A. R. Eckberg, Eastman Kodak Co.
Comdr. E. C. Forsyth, Bureau of Ships
Lt. W. P. Goepfer, Bureau of Aeronautics
S. K. Varnes, E. I. du Pont de Nemours and Co.

This group has prepared the following specifications covering clad materials:

Specifications for:

Corrosion-Resisting Chromium Steel Clad Plate, Sheet, and Strip,
Corrosion-Resisting Chromium-Nickel Steel Clad Plate, Sheet, and Strip, and
Nickel and Nickel-Base Alloy Clad Steel Plate.

These specifications have been approved by the subcommittee and will be submitted to Committee E-10 on Standards.[9]

Subcommittee X on Specifications for Castings (J. J. Kanter, chairman).—The subcommittee met in Buffalo, N. Y., on March 4, 1943. The problem of rewriting the specifications for castings was reviewed in the light of several meetings held on the subject of high-alloy castings under the auspices of the Alloy Castings Institute, the American Petroleum Institute, and A.S.T.M. Committee B-4 on Electrical-Heating, Electrical-Resistance, and Electric-Furnace Alloys. It was the feeling that for the purpose of Committee A-10, the testing requirements being proposed in the specifications for 25 per cent chromium, 12 per cent nickel alloy under consideration by these other groups would not be practical requirements, particularly under war-time conditions. H. S. Avery and R. C. Sutton were appointed as a subgroup to consider the drafting of casting specifications satisfactory to Committee A-10. Examination will be made of a recently issued A.P.I. specification for 25 per cent chromium, 12 per cent nickel alloy and an attempt will be made to harmonize promptly this specification with those prepared by this subgroup for Committee A-10.

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[9] See Editorial Note, p. 107.

Subcommittee XI on Specifications for Tubular Products (J. J. B. Rutherford, chairman).—A joint committee consisting of members from Subcommittee IX on Steel Tubing and Pipe of Committee A-1 and Subcommittee XI of Committee A-10 has been organized to prepare specifications for stainless steel tubular products. It is expected that four specifications will be prepared, use being made of the Specifications for Stainless Steel Tubing published in the Steel Products Manual of the American Iron and Steel Institute. These specifications are being prepared in response to a request from the Administrative Committee of N.E.S.S.

This report has been submitted to letter ballot of the committee which consists of 80 members, 68 members returned their ballots, of whom 67 have voted affirmatively, and 1 negatively.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

H. D. NEWELL,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-10 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:

Corrosion-Resisting Chromium Steel Clad Plate, Sheet, and Strip (A 263 – 43 T),

Corrosion-Resisting Chromium-Nickel Steel Clad Plate, Sheet, and Strip (A 264 – 43 T), and

Nickel and Nickel-Base Alloy Clad Steel Plate (A 265 – 43 T).

These new tentative standards were accepted by Committee E-10 on August 30, 1943, and assigned the A.S.T.M. serial designations indicated above. They appear in the 1943 Supplement to the Book of A.S.T.M. Standards, Part I, pp. 184, 192, and 200.

Committee A-10 also recommended changes in the Emergency Alternate Provisions in Standard Specifications for Corrosion-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip (EA – A 167) and for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (EA – A 240) which were accepted by Committee E-10 on August 30, 1943. The revised emergency provisions were published in ASTM BULLETIN, No. 125, December, 1943, p. 64, under the designations EA – A 167a and EA – A 240a, and have been incorporated in the pink stickers for attachment to the specifications to which they apply.

REPORT OF THE RESEARCH COMMITTEE
ON
FATIGUE OF METALS

During the past year it has not been found feasible to hold a meeting of the Research Committee on Fatigue of Metals. This was due mainly to transportation difficulties and the pressure of war work at practically all testing laboratories.

The cooperative arrangements with *Metals and Alloys* has been continued, although the change of policy of that periodical from the inclusion of short abstracts of a very large number of articles to the publishing of more comprehensive abstracts of a few specially important articles has materially lessened the work of the committee in carrying out their part of this cooperative effort.

During the year a group of committee members visited the Pennsylvania State College and inspected a direct tension-compression testing machine of the resonance type constructed by R. K. Bernhard, also a member of the committee. This was an experimental machine but seemed to give promise of very considerable usefulness.

During the year a project was outlined by the committee at the request of G. F. Jenks, chairman of the A.S.T.M. Committee E-9 on Research, for a rather extensive study of the fatigue strength of screw threads. This project was favorably received by the National Defense Research Council, but, owing to the decision of that body to postpone long-time research projects, it has not been put into operation.

The principal work of the committee during the year has been the continuation of the study of the Effect of Size of Specimens on Fatigue Strength of Three Types of Steel and a Second Progress Report on this investigation is presented as an Appendix to this report.

This report has been submitted to letter ballot of the committee which consists of 24 members; 23 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. F. MOORE,
Chairman.

APPENDIX

SECOND PROGRESS REPORT ON THE EFFECT OF SIZE OF SPECIMEN ON FATIGUE STRENGTH OF THREE TYPES OF STEEL

PREPARED BY H. F. MOORE[1] AND D. MORKOVIN[1]

SYNOPSIS

Further tests on the effect of size on the fatigue strength of notched specimens of two plain carbon steels (S.A.E. 1020 and S.A.E. 1035) and on one heat-treated chromium molybdenum steel (S.A.E. X4130) have been made at the Talbot Materials Testing Laboratory at the University of Illinois. The results of these tests have been correlated with the results of tests of unnotched specimens of the same steels reported last year.[2] The scheme of cutting specimens of different sizes from the bars of steel, and the testing machines used were the same as reported last year.

The tests showed a tendency for the endurance limit of the steels tested to become constant for notched specimens 1 in. in diameter and larger. This was in harmony with the results reported last year for unnotched specimens. It is highly desirable that this result should be checked by tests of still larger specimens along with corresponding tests of smaller specimens, taking great care to reduce variation in surface conditions and differences in strength due to radial variations of strength within the bars of the metal.

Comparisons of theoretical and actual reductions of fatigue strength by notches were made, and it was found that the steels tested came nearer to following the maximum distortion-energy theory (sometimes called the maximum shear strain-energy theory) than to following the common maximum stress theory or maximum shear-stress theory.

Notch sensitivity factors for the different steels were determined by both stress and energy theories and for the different sizes of specimen tested. Curious behavior of the notch sensitivity factors for small sizes of notched specimens was observed, corresponding to marked deviation of their endurance limits from those predicted by the theories.

Considerable use is made of Neuber's theoretical treatment of stress concentration, but his semi-empirical correction for relative size of the stress raiser and grain size, giving "technical stress concentration factor," was found not applicable to the fatigue data herein reported.

A diagram is given with the intention of facilitating approximate estimating of endurance limits of members of any size if the endurance limit of a geometrically similar specimen of the same metal but of a different size is known.

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[1] Research Professor of Engineering Materials, and Instructor in Theoretical and Applied Mechanics, respectively, University of Illinois, Urbana, Ill.

[2] "Progress Report on the Effect of Size of Specimen on Fatigue Strength of Three Types of Steel," H. F. Moore and D. Morkovin, *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 145 (1942).

Scope of Investigation:

The investigation of "size effect" on fatigue strength was planned to cover three main lines of study: (1) effect of size on unnotched, polished specimens, (2) effect of size on notched specimens, and (3) effect of internal stresses in metals studied. The first progress report considered the first of these objectives.[2] This second progress report treats mainly of the second objective. It was hoped that in this second report the magnitude and effect of internal stresses could be considered, but unavoidable delays in securing apparatus for measuring internal strains (from which internal stresses could be computed) have made this impossible at this time. However, it is planned to carry on a study of internal stresses during the coming year.

TABLE I.—HEAT ANALYSES OF THE STEELS USED IN SIZE EFFECT TESTS.

Steel	Chemical Content, per cent						
	Carbon	Manganese	Silicon	Chromium	Molybdenum	Sulfur	Phosphorus
S A.E. 1020	0.22	0.46	0.20	....	....	0.033	0.014
S.A.E. 1035	0.34	0.78	0.24	....	....	0.028	0.016
S.A.E. X4130	0.31	0.55	....	1.08	0.20	0.019	0.017

TABLE II.—MECHANICAL PROPERTIES OF THE STEELS USED IN SIZE EFFECT TESTS.

Steel	Yield Strength, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Average Brinell Hardness Number
S.A.E. 1020	32 300[a]	62 000	28.0	59	125
S.A.E. 1035	47 200[a]	87 600	25.0	50	175
S.A.E. X4130	115 600[b]	141 800	17.0	60	300

[a] Yield point by halt of pointer, rate of strain below yield point 0.042 per cent per min.

[b] Yield strength by offset of 0.2 per cent, rate of strain below yield strength 0.042 per cent per min.

Metals Tested and Testing Machines:

The steels tested were from the same bars as those reported in 1942: namely, (1) 15 bars each 3½ in. in diameter and 20 ft. long of an S.A.E. 1020 carbon steel hot-rolled and furnished by the Carnegie-Illinois Steel Corp., (2) 16 bars each 3½ in. in diameter and 19 ft. long of an S.A.E. 1035 steel hot-rolled and furnished by the Carnegie-Illinois Steel Corp., and (3) 11 bars each 3 in. in diameter and in lengths varying from 17 to 21 ft. long of S.A.E. X4130 steel heat treated to approximately 300 Brinell hardness and furnished by the Timken Roller Bearing Co. Table I gives the chemical composition and Table II the mechanical properties of the steels tested.

Testing machines were the same machines used in making the tests of the unnotched bars which were reported in 1942.[2] The type of machine is shown in Fig. 1.

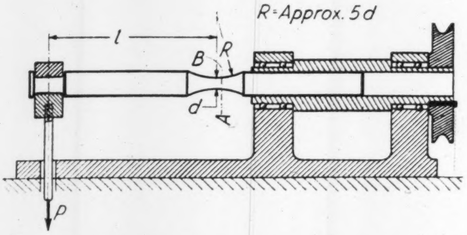

FIG. 1.—Diagram of Typical Test Specimen (Unnotched) and Rotating-Cantilever Testing Machine.

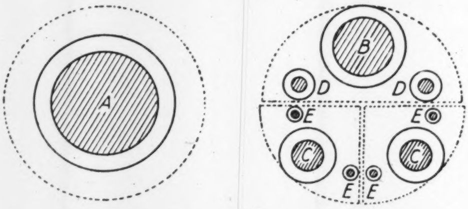

FIG. 2.—Location of Specimens in the Rolled Bars.

A—1½, 1¾, and 1⅞ in. specimens
B—1-in. specimens
C—½-in. specimens
D—¼-in. specimens
E—⅛-in. specimens

Fatigue Specimens:

In cutting fatigue specimens from the rods as furnished, the axis of the largest specimen was the same as the axis of the bar. The other specimens were cut so that each specimen had one inner "extreme fiber" at the critical section at a distance from the axis of the bar approximately equal to the critical-section radius of the largest specimen. The location of the specimens in the bars is shown in Fig. 2.

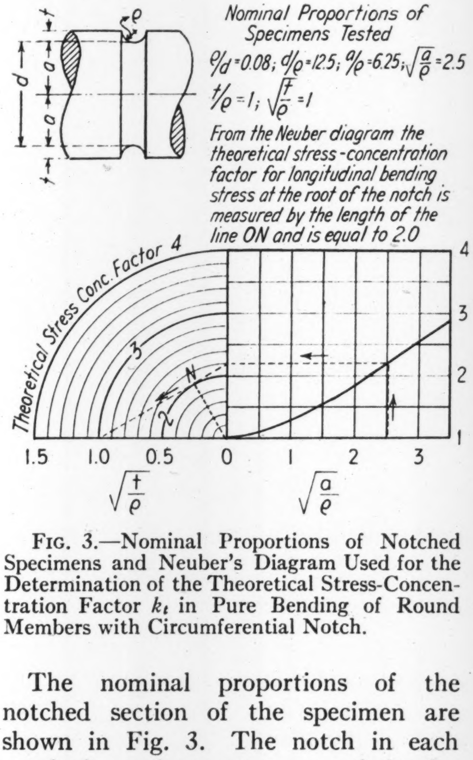
ρ
t
a
a
d
t
Nominal Proportions of Specimens Tested
ρ/d = 0.08; d/ρ = 12.5; a/ρ = 6.25; √(a/ρ) = 2.5
t/ρ = 1; √(t/ρ) = 1
From the Neuber diagram the theoretical stress-concentration factor for longitudinal bending stress at the root of the notch is measured by the length of the line ON and is equal to 2.0
Theoretical Stress Conc. Factor 4
3
2
N
4
3
2
1
1.5
1.0
0.5
0
1
2
3
√(t/ρ)
√(a/ρ)

FIG. 3.—Nominal Proportions of Notched Specimens and Neuber's Diagram Used for the Determination of the Theoretical Stress-Concentration Factor k_t in Pure Bending of Round Members with Circumferential Notch.

The nominal proportions of the notched section of the specimen are shown in Fig. 3. The notch in each notched specimen was a semi-circular groove with a radius equal to 0.08 times the minimum diameter of the specimen at the root of the notch.

Figure 3 shows also the Neuber diagram[3] for theoretical bending stress concentration at the root of a groove of hyperbolic profile with radius of curvature ρ, depth of notch t, and minimum diameter of the specimen at the root of notch $2a$. It was assumed that the stress concentration factor at the root of a semi-circular groove does not differ appreciably from the stress concentration factor at the root of a hyperbolic groove of the same radius of curvature ρ and depth t. Using the diagram in Fig. 3 as indicated by the arrows, the value of the theoretical stress concentration factor k_t at the root of the groove of the proportions common to all notched specimens tested was found to be 2.0. This theoretical stress concentration factor k_t is the ratio $\frac{s_1}{s_n}$, in which s_1 denotes the maximum longitudinal stress at the root of the notch (as calculated by Neuber), and s_n denotes the nominal longitudinal stress in the same fiber as computed by the ordinary flexure formula.

Theories of Failure Applied to Fatigue of Notched Specimens:[4]

Let the endurance limit stress as determined by tests of unnotched polished specimens be called s_e. The "strength-reduction factor" or "effective stress concentration factor in fatigue" of notched specimens is then the ratio $\frac{s_e}{s_n}$. The values of this ratio obtained by experiment are called k_f (experimental fatigue strength-reduction factor). The values of the ratio $\frac{s_e}{s_n}$ predicted by the various theories of failure are called k_t' (theoretical strength-reduction factor).

If the maximum normal stress theory

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[3] H. Neuber, "Kerbspannungslehre," Julius Springer, Berlin (1937), Fig. 104.

[4] The treatment given here follows quite closely that of R. E. Peterson in a paper presented before the Society for the Promotion of Engineering Education, June 2, 1941, at the University of Michigan. A description of the various theories is available in several textbooks, see for example S. Timoshenko, "Strength of Materials," Part II, D. Van Nostrand Co., Inc., New York, N. Y., p. 711.

of failure is applied to fatigue of notched specimens, it can be expressed as follows: The fatigue failure will not occur in the notched specimen if the maximum normal stress s_1 does not exceed the normal stress s_e in the highest stressed fiber of an unnotched fatigue specimen at its endurance limit. Written in symbols this is

$$s_1 = s_e \dots\dots\dots\dots (1)$$

or, by definition of k_t,

$$k_t s_n = s_e \dots\dots\dots\dots (2)$$

Transposing,

$$k_t = \frac{s_e}{s_n} \dots\dots\dots\dots (3)$$

or, by definition of k_t'

$$k_t = k_t' \dots\dots\dots\dots (4)$$

If the maximum shear stress theory of failure is applied to fatigue of notched specimens, the following statement can be made: The fatigue failure will not occur in the notched specimen if the maximum shear stress $\frac{s_1}{2}$ at the root of the notch in the notched specimen does not exceed the maximum shear stress in the highest stressed (surface) fiber $\frac{s_e}{2}$ in the unnotched polished specimen at the endurance limit. In symbols this statement reads:

$$\frac{s_1}{2} = \frac{s_e}{2} \dots\dots\dots\dots (5)$$

or

$$s_1 = s_e \dots\dots\dots\dots (6)$$

Equation 6 is the same as Eq. 1 and leads therefore also to Eq. 4.

From Neuber's formulas and diagrams[3] there can be determined not only the longitudinal stress s_1 at the root of the notch, but also the circumferential stress s_2 and the radial stress s_3. The circumferential stress is given as $s_2 = \mu(s_1 - s_n)$. In this, s_1 denotes the longitudinal stress at the bottom of the notch as given by the Neuber diagram (Fig. 3); s_2 denotes the circumferential stress at the root of the notch; s_n denotes the stress computed by the ordinary flexure formula; and μ denotes Poisson's ratio for the metal.

The radial stress, s_3, at the root of the notch is zero at the surface. Now s_n can be expressed as $\frac{s_1}{k_t}$ by definition of k_t. For the specimens tested $s_2 = \mu\left(s_1 - \frac{s_1}{k_t}\right) = \frac{\mu s_1}{2}$, because $k_t = 2.0$. Then $\frac{s_2}{s_1} = \frac{\mu}{2}$ for the specimens tested,[5] or denoting $\frac{\mu}{2}$ by c: $s_2 = cs_1$.

The maximum shear strain-energy theory (sometimes called the distortion-energy theory) of Huber, von Mises and Hencky seems to fit best the test results of recent investigators. This theory is based on the shear or distortion energy which is found by subtracting the energy due to volume change from the total strain energy per unit volume. If we consider fatigue failure, the theory may be stated as follows: Fatigue fracture will not occur under biaxial or triaxial stress in a notched specimen if the *distortion-energy per cubic inch* of the highest stressed element does not exceed that in an unnotched fatigue specimen of the same metal under uniaxial stress at its endurance limit; or expressed as a formula,

$$\frac{(1+\mu)}{6}\left[(s_1 - s_2)^2 + (s_2 - s_3)^2 + (s_3 - s_1)^2\right] = \frac{(1+\mu)}{3} s_e^2 \dots (7)$$

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[5] In the actual test results slight corrections have been made for variations of t and ρ from the nominal values.

in which, for a round notched specimen s_1 denotes longitudinal stress, s_2 denotes circumferential stress, and s_3 denotes radial stress.

Now from Neuber we have seen that $s_3 = 0$, and for the notched specimens tested in this investigation $s_2 = \frac{\mu}{2}s_1 = cs_1$.

Values of k_t' and k_t for the specimens tested are as follows:

Theory	k_t'	k_t
Maximum Normal Stress Theory and Maximum Shear Stress Theory	2.00	2.00
Distortion-Energy Theory, μ = 0.25	1.886	2.00
Distortion-Energy Theory, μ = 0.27	1.880	2.00
Distortion-Energy Theory, μ = 0.30	1.870	2.00

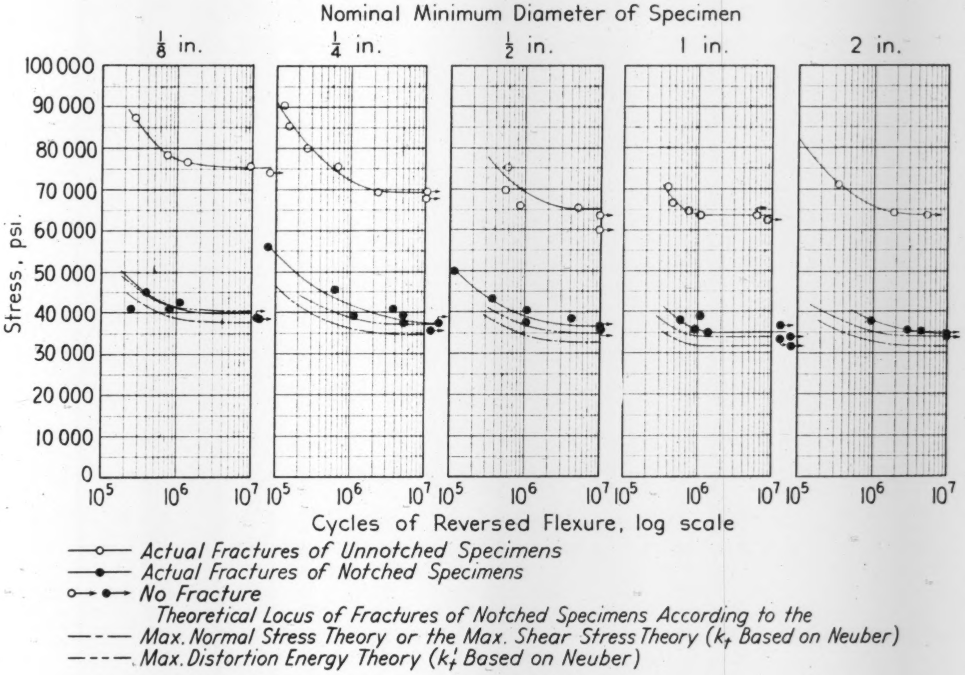

FIG. 4.—*S-N* Diagrams for S.A.E. X4130 Steel.

Then Eq. 7 becomes

$$s_1^2[(1 - c)^2 + c^2 + 1] = 2s_e^2 \quad \ldots (8)$$

$$s_1^2(2 - 2c + 2c^2) = 2s_e^2 \quad \ldots (9)$$

$$s_1\sqrt{1 - c + c^2} = s_e \quad \ldots (10)$$

Dividing both sides of Eq. 10 by s_n and recalling that $\frac{s_1}{s_n} = k_t$ and $\frac{s_e}{s_n} = k_t'$

$$k_t\sqrt{1 - c + c^2} = k_t' \quad \ldots (11)$$

In this investigation $k_t = 2$ and thus, according to distortion-energy theory,

$$k_t' = 2\sqrt{1 - c + c^2} \quad \ldots (12)$$

The values of k_t' for a range of Poisson's ratio, μ, as wide as is usually found for steel shows a very slight variation and the value 0.30 is used in this report.

Polishing of Specimens:

The unnotched specimens of 2, 1, $\frac{1}{2}$, and $\frac{1}{4}$-in. sizes and notched ones of 2-in. size were polished with Metalite cloth grit 120 and finished with Aloxite cloth grit 240, with oil. The unnotched specimens of $\frac{1}{8}$-in. size were finished with

Emery paper grit No. 2/0, with oil. The cloth or paper was held on a mandrel and both specimen and mandrel rotated. Notches in specimens of sizes 1, $\frac{1}{2}$, $\frac{1}{4}$, and $\frac{1}{8}$-in. were polished with a rotating soft copper wire of proper diameter, flooded with oil with fused alumina size 240 (alundum grain), and finished in the same manner with silicon carbide grain No. FFF. The axis of the swiftly rotating mandrel was held at approximately 90 deg. with the axis of the slowly rotating specimen.

Results of Fatigue Tests:

Figures 4, 5, and 6 show *S-N* diagrams for both the unnotched bars and the notched bars of the steels tested. For comparison with actual results, these figures also show two lines indicating theoretical *S-N* diagrams for notched bars based on: (1) the maximum tensile stress theory (and the maximum shear-stress theory as well), and (2) on the maximum distortion-energy theory (sometimes called the maximum shear strain-energy theory).

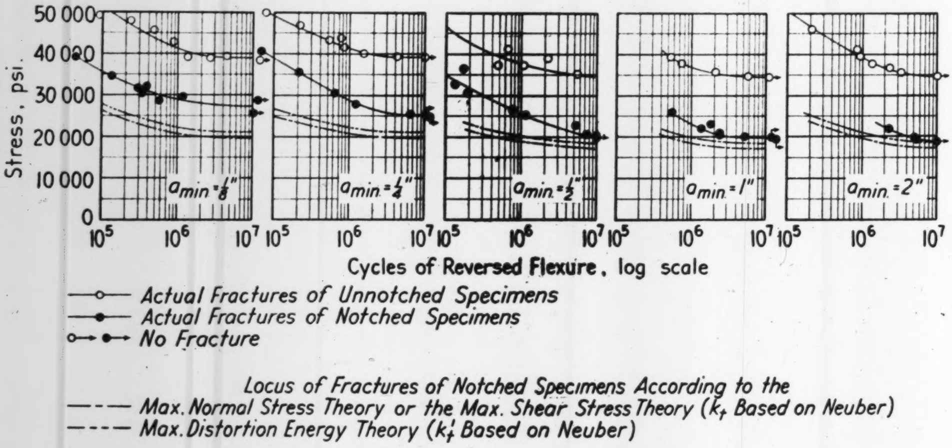

FIG. 5.—*S-N* Diagrams for S.A.E. 1035 Steel.

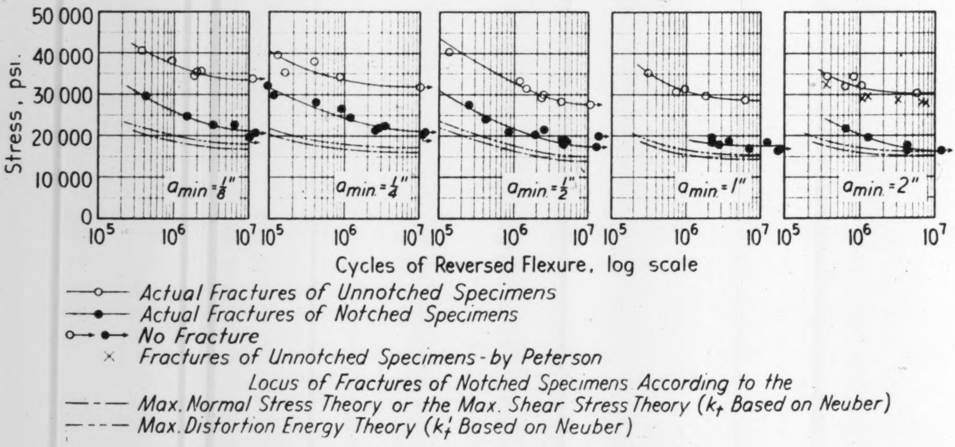

FIG. 6.—*S-N* Diagrams for S.A.E. 1020 Steel.

The results for the unnotched specimens were those reported in the 1942 Progress Report, except that examination of the data indicated that the correction for variation in hardness of steel at different distances from the axis of the specimens was very small due to the fact that different specimens were located in the bar with a critical "fiber" on the most stressed surface at very nearly the same distance from the axis of the bar; so that this correction seemed unnecessary, and has not been made either for the results of tests on unnotched bars nor for results of tests on notched bars.

TABLE III.—STRENGTH REDUCTION FACTORS FOR SEMICIRCULAR NOTCHES WITH RADIUS OF CURVATURE 8 PER CENT OF THE CRITICAL DIAMETER OF SPECIMEN.

Steel	Critical Diameter of Specimen, in.	Strength Reduction Factor (k_t)				
		By Fatigue Test (k_f)	By Maximum Tensile Stress Theory or Maximum Shear Stress Theory		By Shear Strain-Energy Theory (Sometimes called Distortion-Energy Theory)	
			Neuber	Peterson	Neuber	Peterson
S.A.E. 1020	0.125	1.55	2.00	1.90	1.87	1.75
	0.250	1.59	2.00	1.90	1.87	1.75
	0.500	1.63	2.00	1.90	1.87	1.75
	1.000	1.64	2.00	1.90	1.87	1.75
	1.875	1.64	2.00	1.90	1.87	1.75
S.A.E. 1035	0.125	1.54	2.00	1.90	1.87	1.75
	0.250	1.62	2.00	1.90	1.87	1.75
	0.500	1.73	2.00	1.90	1.87	1.75
	1.000	1.77	2.00	1.90	1.87	1.75
	1.875	1.77	2.00	1.90	1.87	1.75
S.A.E. X4130	0.125	1.93	2.00	1.90	1.87	1.75
	0.250	1.88	2.00	1.90	1.87	1.75
	0.500	1.82	2.00	1.90	1.87	1.75
	1.000	1.82	2.00	1.90	1.87	1.75
	1.750	1.82	2.00	1.90	1.87	1.75

On Fig. 6 are plotted results of tests on large specimens of S.A.E. 1020 steel made by R. E. Peterson at the Westinghouse Research Laboratories. These results are indicated by crosses. The specimen used in the Westinghouse tests had a tapered section with a practically uniform stress over several inches of length. This may explain the slightly lower values obtained.

Table III shows strength-reduction factors and Fig. 7 shows endurance limits for the various sizes of specimens tested. It will be noted that for both the unnotched and the notched specimens of all three metals the maximum size effect seemed to have been reached with the 1-in. specimens, and that no additional reduction of fatigue strength is shown by the tests of the larger specimens. This result is very important, and it seems highly desirable that it should be checked in the near future by fatigue tests of specimens of various sizes, including some specimens larger than 2 in. in diameter. In such tests great care should be used in keeping surface conditions as nearly alike as possible and in cutting specimens from the bars of metal so that each specimen has one inner "fiber" at the critical section at approximately a constant distance, presumably a distance equal to the critical-section radius of the largest specimen.

In Fig. 7 the values for endurance limits given by the maximum stress theories and the maximum shear strain-energy (or maximum distortion-energy) theory are shown by broken lines. These values were obtained by dividing ordinates of the curves for actual test results by the theoretical strength-reduction factors, k'_t. The actual (test) strength-reduction factors obtained from the test results, k_f, as well as the theoretical strength-reduction factors based on the different theories are given in Table III. The only difference between the results under the heading "Neuber" and those under the heading "Peterson" is the following.

The theoretical strength-reduction factors of Peterson[4] are based on photoelastic tests by Frocht on flat specimens with semi-circular notches. For determining the theoretical strength-reduction factors marked "Neuber," use was made of the theoretical results determined from the Neuber diagram shown in Fig. 3. The actual strength-reduction factors k_f were obtained by dividing the endurance limit of unnotched specimens by the corresponding endurance limit of the notched specimens.[6]

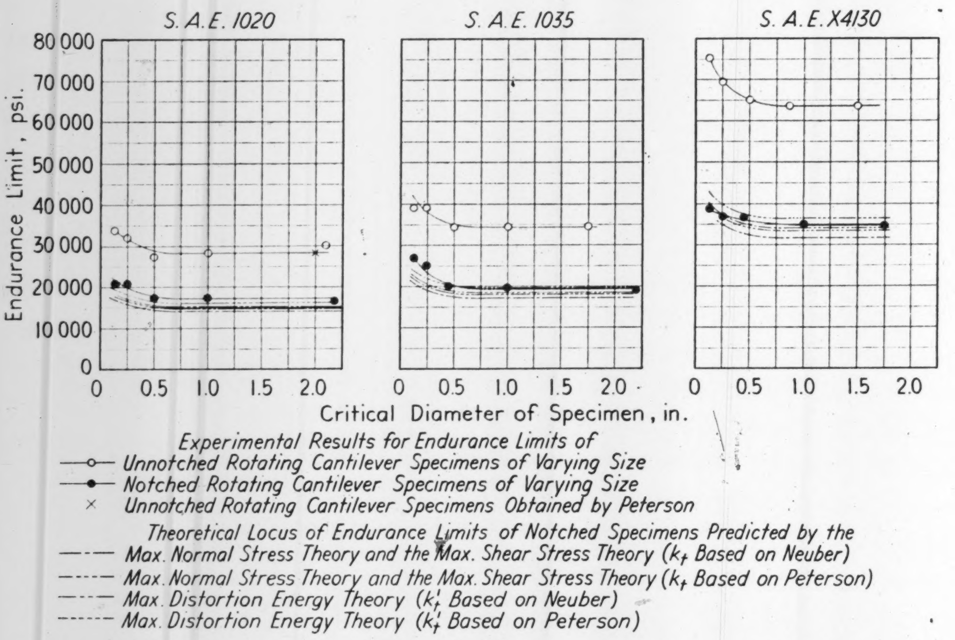

FIG. 7.—Effect of Size of Specimen Upon the Endurance Limit in Reversed Bending of notched and Notched Rotating-Cantilever Specimens for Three Representative Kinds of Steel.

Comparison of experimental results with results predicted by various theories of failure.

It is seen that for the S.A.E. 1020 steel the actual endurance limits of notched specimens are slightly greater, by an approximately constant amount for all the sizes, than the theoretical values predicted by any of the theories of failure. The maximum distortion-energy theory is seen to come closer to the actual test results than the maximum stress theories.

In the case of the S.A.E. 1035 steel, the endurance limits of notched specimens practically coincide with those predicted by the maximum distortion-energy (using Peterson's k_t') theory for specimens $\frac{1}{2}$ in. in diameter and larger. The endurance limits of small specimens are considerably higher than any of the theories predict.

In the case of the S.A.E. X4130 steel, the endurance limits of notched specimens lie between the two curves predicted by the maximum distortion-energy theory, using Peterson's and Neuber's k_t, respectively. The endur-

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[6] The probable "notch effect" of the reduced portion of the "unnotched" specimens was investigated by the use of the Neuber diagram, Fig. 3. For the unnotched specimens $\sqrt{\frac{a}{\rho}} = \sqrt{\frac{0.05d}{5d}} = 0.316$ (see Fig. 1) and the maximum value of $\sqrt{\frac{t}{\rho}}$ was found to be 0.04. Using the diagram, the value of k_t was found to be slightly less than 1.1 and the value of k_t' by the maximum distortion-energy theory would be still smaller.

TABLE IV.—NOTCH SENSITIVITY FACTORS (*q*) FOR SEMICIRCULAR NOTCHES WITH RADIUS OF CURVATURE OF NOTCH 8 PER CENT OF THE CRITICAL DIAMETER OF SPECIMEN

$$q = \frac{k_f - 1}{k_t' - 1}$$

Steel	Critical Diameter of Specimen, in.	Notch Sensitivity Factor (*q*)			
		By Maximum Tensile Stress Theory or Maximum Shear Stress Theory		By Shear Strain-Energy Theory (Sometimes called Distortion-Energy Theory)	
		Neuber	Peterson	Neuber	Peterson
S.A.E. 1020	0.125	0.55	0.61	0.63	0.74
	0.250	0.59	0.66	0.68	0.79
	0.500	0.63	0.70	0.73	0.85
	1.000	0.64	0.71	0.74	0.86
	1.875	0.64	0.71	0.74	0.86
S.A.E. 1035	0.125	0.54	0.60	0.62	0.72
	0.250	0.62	0.69	0.72	0.83
	0.500	0.73	0.81	0.84	0.98
	1.000	0.77	0.86	0.89	1.03
	1.875	0.77	0.86	0.89	1.03
S.A.E. X4130	0.125	0.93	1.03	1.07	1.25
	0.250	0.88	0.98	1.01	1.18
	0.500	0.82	0.91	0.94	1.10
	1.000	0.82	0.91	0.94	1.10
	1.750	0.82	0.91	0.94	1.10

ance limits of small specimens ($\frac{1}{4}$ and $\frac{1}{8}$-in.) are smaller than the distortion energy theory predicts. The use of Neuber's values k_t lead to fair approximations of the test values for all sizes of specimens tested.

From the above observation on Fig. 7 and Table III, the following conclusion may be drawn: For the metals tested the maximum distortion-energy theory fits the actual fatigue test results of notched specimens better than the maximum normal stress theory or the maximum shear stress-theory, for large specimens. However, there are apparent tendencies of the small specimens to behave differently from what any of the theories predict. These tendencies, which show opposite directions in the case of different steels (compare notched specimens of S.A.E. 1035 and S.A.E. X4130 on Fig. 7) are probably another manifestation of the same phenomenon

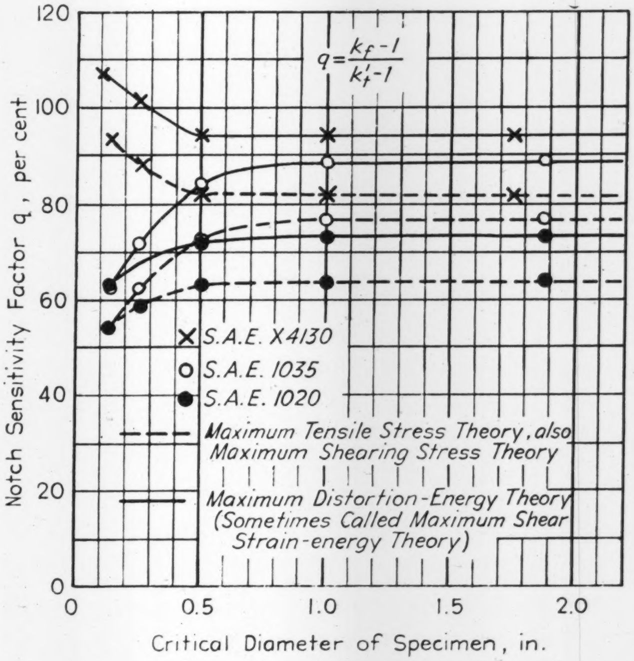

FIG. 8.—Notch-Sensitivity Factors in Reversed Bending Fatigue of Rotating-Cantilever Specimens and the Effect of Size of Specimen.

called here "size effect." Peterson's[4] attempt to explain similar tendencies by assuming that the failure may be considered to occur when the endurance limit is reached at a constant small depth beneath the surface does not seem to be very successful. There are the following alternatives:

(*a*) Distortion-energy theory of fatigue failure is the correct statistical explanation of fatigue failure of notched specimens. The stress concentration factor, however, is some function of the relative size of stress raiser and grain size.

(*b*) None of the theories of failure which were considered apply, and in fatigue failure there are factors unaccounted for by these theories.

It should be mentioned in connection with alternative (*a*) that Neuber (p. 144 and Fig. 106)[3] tries to take into account the influence upon the stress concentration factor of the relative size of the stress raiser (namely, the radius of curvature of notch, ρ, and something similar to grain size, ρ'. An attempt has been made to check his "technical stress concentration factor" with the results of the tests reported on here, but no agreement was found. It seems that Neuber's semi-empirical method of introducing ρ' does not explain this phenomenon in fatigue.

Notch Sensitivity of Metals Tested:

The notch sensitivity of a metal has been defined as the ratio of actual loss of fatigue strength due to the notch to the theoretical loss of fatigue strength. That is, if q represents notch sensitivity,

$$q = \frac{k_f - 1}{k_t' - 1}$$

Table IV and Fig. 8 show variations of the notch sensitivity with diameter of specimen for the three metals tested. It is noted, as might be expected from the endurance limit curves, that the notch sensitivity observed during the test remains constant for sizes of 1 in. and larger for the specimens tested.

As the diameter of the specimen decreases below $\frac{1}{2}$ in., the notch sensitivity no longer remains constant. For the two plain carbon steels the notch sensitivity decreased with the decrease in size, whereas for the heat-treated alloy steel it increased with the decrease in size.

This phenomenon seems to be the same that was observed above when the two alternative conclusions about theories of fatigue failure were discussed. It is conceivable that the stress concentration factor is a function of the relative size of the stress raiser and some physical dimension of particles of the material, say, of the order of magnitude of grain size.

It is not clear why the notch sensitivity factor in the case of the plain carbon steels should behave in a diametrally opposite way to that for the heat-treated alloy steel in specimens of small size. Possibly one line of investigation might be this: Could the machining and polishing of the grooves affect microscopic stress concentrations on the surface of the grooves of different sizes and kinds of steel differently? All three metals and all sizes were machined and polished in the way described above under "Polishing of Specimens." The ratio of the size of the polishing scratches to the specimen size has not as yet been investigated. The different machinability of the steels might cause microscopic stress concentrations on the surface that might be of different relative size in materials of different machinability.

Effect of Size on Endurance Limit on a Percentage Basis:

As is apparent from Fig. 7, the increase of size beyond about 1 in. in

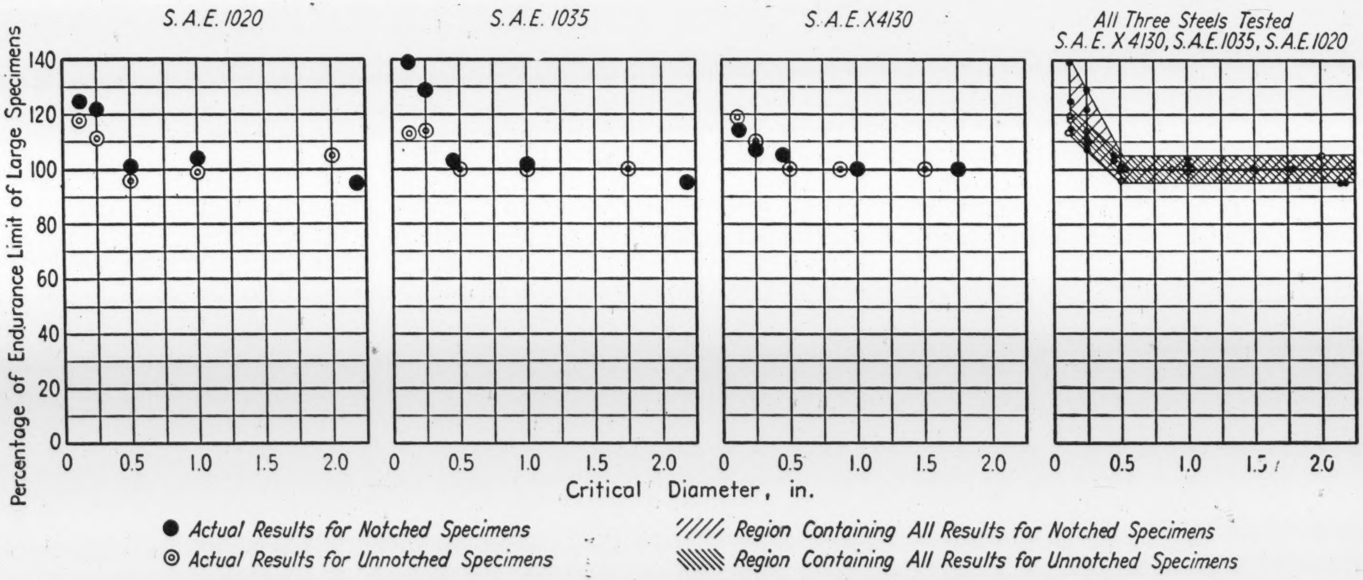

FIG. 9.—Endurance Limits in Reversed Bending of Unnotched and Notched Rotating-Cantilever Specimens of Various Sizes, Expressed as Percentages of the Average Endurance Limits of Large Specimens (Diameter greater than 1 in.).

diameter does not seem to reduce the endurance limit of either unnotched or notched specimens any further. This conclusion should be checked by fatigue tests on specimens of still larger size (keeping all other factors constant); but for the steels tested it seems to be fairly well substantiated by the data reported here. For purposes of comparison, therefore, the endurance limit of large specimens of each steel was taken as the basis and the endurance limits for specimens of all sizes were recalculated as percentages of this "endurance limit of large specimens" (1 in. or larger). The results are shown in Fig. 9. In the composite diagram of Fig. 9, the results are given for both the notched and unnotched specimens of all three kinds of steel, expressed as percentages of the corresponding endurance limit of large specimens.

The useful purpose that this combined diagram in Fig. 9 might serve in design may be illustrated by the following numerical example: Suppose it is desired to estimate a safe value of the endurance limit of a 2-in. diameter notched rotating-beam steel member. From a series of $\frac{1}{4}$-in. specimens geometrically similar to the large member, made from the same steel from which the 2-in. member is to be machined, an endurance limit of, say, 30,000 psi. was obtained. The ordinate (corresponding to $\frac{1}{4}$-in. specimen) of the top margin of the shaded region in Fig. 9 is about 130 per cent. The ordinate at the bottom edge of the shaded region at 2 in. is about 95 per cent. Therefore, a safe estimate is that the endurance limit of the 2-in. specimen will not be below $30{,}000 \times \frac{95}{130} = 22{,}000$ psi. For an unnotched specimen, the differently shaded region, including all results for the unnotched specimens, may be used in a similar way.

Acknowledgments:

The work of the past year could not have been carried on had it not been for the generous financial support of the Research Committee of the American Society for Testing Materials, and grateful acknowledgment is here made for their grants from the society research fund.

Acknowledgments are made to the Westinghouse Laboratories for the tests they have made and are making on 2-in. specimens.

The work in the Talbot Laboratory was carried on as a part of the regular work of the University of Illinois Engineering Experiment Station, Dean M. L. Enger, Director and of the Department of Theoretical and Applied Mechanics, Prof. F. B. Seely, Head.

DISCUSSION

Mr. G. W. Stickley[1] (*presented in written form*).—This investigation of a wide range of sizes of specimens is very interesting, especially since it was possible to prepare all of the specimens from the same size and lot of rod. The authors were fortunate in this respect, and as a result the value of their tests has been greatly increased. Consideration still had to be given, of course, to variations in properties from the outside to the center of the rod, for they report that this variation in hardness was 10 to 15 per cent. Therefore, the locations of the various sizes of specimens were carefully chosen. Even with the plan that was used, however, it still is possible that it may not be correct to attribute all of the variations in results of the fatigue tests to differences in size of specimen. Any such question probably could be answered by making several series of fatigue tests using one of the smaller sizes of specimens, with the specimens in the different series being taken at different locations between the axis and the surface of the rod. Such tests would seem desirable to confirm the conclusion that a safe value for endurance limit of a steel member of large size can be estimated from tests of relatively small specimens, as has been suggested by the authors.

Messrs. O. J. Horger[2] **and H. R. Neifert.**[2]—Under the subject heading of notch sensitivity of metals tested and in Fig. 8, the authors suggest that the condition of surface finish of the test specimens may have some influence upon the diametrically opposite manner in which the notch sensitivity of the alloy steel behaves in comparison with plain carbon steels. Surface finish measurements were made on a number of specimens furnished by the authors to give data on this question. Brush analyzer records were made circumferentially on both notched and unnotched specimens in the region of maximum bending stress as shown in the accompanying Fig. 1.

TABLE I.—PROFILOMETER READINGS OF SURFACE FINISH.

Steel	Notched Specimens (Circumferential Trace)				Unnotched Specimens							
					Same values for longitudinal and circumferential trace				Longitudinal trace			
	2 in. diameter		1 in. diameter		2 in. diameter		1 in. diameter		½ in. diameter		¼ in. diameter	
	Specimen	Finish, μ in.	Specimen	Finish, μ in.	Specimen	Finish, μ in.	Specimen	Finish, μ in.	Specimen	Finish, μ in.	Specimen	Finish, μ in.
X-4130	XI	10–12	XI-2	8–10	XI	17–20	XE-1	16–18	XE	9–12	XE-2	10–13
1020	KK	9–13	KK-2	8–10	KK	13–15	LL-1	13–15	PP	10–13	MM-2	9–12
1035	C	12–15	D-1	8–11	A	10–11	A-1	11–13	A-1	10–13	B-2	7–10

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[1] Research Metallurgical Engineer, Aluminum Company of America, Research Laboratories, New Kensington, Pa.

[2] In Charge of Railway Engineering and Research, and Engineer, respectively, Research Laboratory, The Timken Roller Bearing Co., Canton, Ohio.

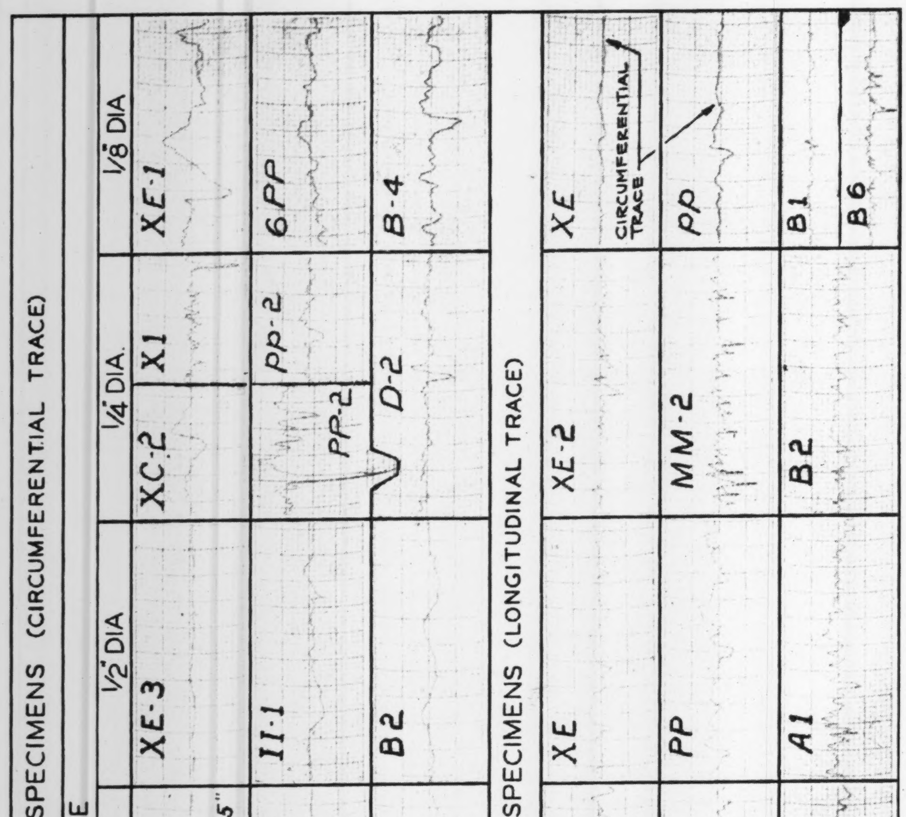

FIG. 1.—Brush Surface Analyzer Record of Surface Finish.
Magnification: Vertical (× 4000); Horizontal (× 80)

The profilometer instrument was also used to obtain readings circumferentially although on some unnotched specimens both longitudinal and circumferential traces were made as given in the accompanying Table I.

These studies of surface finish do not appear to explain this divergence in notch sensitivity found between the carbon and alloy steels in sizes up to ½ in. in diameter. Other investigators as well as the authors have made fatigue tests different from those reported here which should be given the same type of analysis as shown in Fig. 8. Such a study would indicate whether the findings of the authors were general or only a special case. The discussors also wish to point out that if both plain carbon steels had the same hardness as the alloy steel tested then the diametrically opposite paths taken by the notch sensitivity curves in Fig. 8 may be changed.

Referring to Fig. 7, it is apparent that the endurance limit, for both notched and unnotched specimens, begins to flatten off at about ½ in. in diameter. It would be very dangerous to infer that the endurance limit does not change for larger diameter members than the 2 in. shown. Proof of this statement may be based on fatigue tests of large members which the discussors are making at this time but which are not completed. Relatively little laboratory data are available on large sections but the designer knows from experience that lower allowable stresses are more necessary for large than for small sections. One finding which the discussors have made from laboratory fatigue tests on large sections is that there is considerable scatter in results and much more than experienced on small specimens. Besides being a fatigue problem, the study of large sections also becomes a statistical one.

MESSRS. H. F. MOORE[3] AND D. MORKOVIN[3] (*authors' closure, by letter*).—The authors wish to thank the discussors of this paper for emphasizing the necessity of more tests on specimens larger than 2 in. in diameter before accepting as final the results of the tests reported in this paper, which show no further "size effect" for specimens more than 1 in. in diameter. The authors stated this necessity in the paper, and they are glad to see it further emphasized in the discussion.

Mr. Stickley's suggestion of checking the Brinell hardness surveys of steel at different distances from the axis of the bar by means of fatigue tests of small specimens is a good one, and one it is hoped to carry out.

The authors wish to thank Messrs. Horger and Neifert for their studies of surface finish, which have added to the data available on the effect of surface finish, and which seem to confirm the conclusion rather generally reached that beyond a certain refinement of surface finish, say, about that of a finish with 00 or 000 emery cloth, further benefit to the fatigue strength is slight. The authors interpret the last remark of Messrs. Horger and Neifert that, in the case of large members, a factor of "probability of occurrence of a defect of critical size" becomes effective, making the failures of large members a statistical phenomenon. A "probability of occurrence of a defect of critical size" factor was looked for by the authors in their quest of all the factors that may contribute to the size effect, but this probability factor was not found to be effective in the case of sizes tested by the authors.

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[3] Research Professor of Engineering Materials and Instructor in Theoretical and Applied Mechanics, respectively, University of Illinois, Urbana, Ill.

It may be interesting to summarize briefly the work done in this regard since the report was given.

Testing of specimens subjected to a vacuum anneal at 1500 F. (which should have removed most of the residual strains, including those due to machining) still showed size effect of the same order of magnitude as found in unannealed specimens.

The work done by Messrs. Horger and Neifert (see above) seems to show that the polishing scratches were of about the same dimensions on all of the specimens of different sizes. That means that the small specimens had notches of relatively (to the dimension of specimen itself) more gently rounded or "milder" profiles than the large specimens. Calculations are in process at the present time to see how much of the size effect can be due to this factor.

REPORT OF JOINT RESEARCH COMMITTEE
ON
EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS

Progress Report to the Sponsor Societies

During the past year, the Joint Research Committee on Effect of Temperature on the Properties of Metals, under the sponsorship of The American Society of Mechanical Engineers and the American Society for Testing Materials, held one meeting on June 29, 1943, in Pittsburgh, Pa., prior to the presentation of this report at the Annual Meeting of the A.S.T.M.

Finances:

There appears in Table I a summary of the administration of committee funds during the period of May 1, 1942, to May 31, 1943.

TABLE I.—SUMMARY OF RECEIPT AND EXPENDITURES OF COMMITTEE FUNDS.

Balance as of April 30, 1942, shown in 1942 Report			$10 459.69
	Receipts		
1942			
June	The Engineering Foundation	$250.00	
September	Interest on funds (10/1/41 to 9/30/42)	41.67	
	Total Receipts		291.67
			$10 751.36
	Expenditures		
1942			
June	Battelle Memorial Institute (Project No. 18)	$386.35	
August	Ogilvee Press—letterheads	7.83	
1943			
May	Massachusetts Inst. of Technology (Project No. 10)	400.00	
	Total Expenditures		$794.18
Balance on hand, May 31, 1943			$9 957.18

Organization and Membership:

R. H. Aborn of the U. S. Steel Research Laboratories resigned as a member of the committee, and has been replaced by R. F. Miller of the same organization, who has served as alternate for Mr. Aborn for a number of years.

J. H. Romann, Consultant for Tube Turns, Inc., was appointed to membership on the committee during the year.

Research Projects:

There follows a summary of progress made in the various research projects during the year ending June, 1943.

Project No. 18 on Effect of Variables on the High-Temperature Properties of Metals (H. W. Gillett, chairman).—This project has continued to be inactive during the year due to the fact that the apparatus and personnel utilized in this project are assigned to other important studies connected with the war effort.

Project No. 25 on Comparison of Short-Time Test Methods (J. S. Worth, chairman).—The studies involved in this project are nearing completion. The work has been delayed because the personnel dealing with it are occupied by important matters connected with the war effort.

Project No. 26 on Test Methods (J. W. Bolton, chairman).—Last year, the committee recommended that the Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 - 37 T) be revised and adopted as a standard Recommended Practice for Short-Time Elevated-Temperature Tension Tests of Metallic Materials.

Inadvertently, this recommendation was not submitted to letter ballot of the Society subsequent to the 1942 Annual Meeting, and the recommendation to revise Tentative Methods E 21 was accordingly submitted to Committee E-10 on Standards. The revised tentative standard was accepted by the Standards Committee on January 5, 1943, and published under the designation E 21 - 42 T.[1]

The committee now recommends that the original intent of its recommendation be carried out and that the Tentative Recommended Practice for Short-Time Elevated-Temperature Tension Tests of Metallic Materials (E 21 - 42 T)[2] be adopted as standard. This recommendation has been approved by letter ballot vote of the joint committee as follows: From a membership of 25, 25 members returned their ballots, of whom 23 have voted affirmatively, 0 negatively, and 2 members marked their ballots "not voting."

Respectfully submitted on behalf of the Joint Research Committee,

N. L. MOCHEL,
Chairman.

H. J. KERR,
Vice-Chairman.
J. W. BOLTON,
Secretary.

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[1] 1942 Book of A.S.T.M. Standards, Part I, p. 1577.

[2] This recommended practice was adopted as standard by the Society and appears in the 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 104.

REPORT OF JOINT COMMITTEE
ON
FILLER METAL

The Joint A.W.S.-A.S.T.M. Committee on Filler Metal, organized as the best means of correlating the mutual interests of the American Welding Society and A.S.T.M. in matters relating to standards for filler metal used in the various welding processes and as applied to both ferrous and non-ferrous metals and alloys, held its organization meeting in Atlantic City, N. J. on June 22, 1942.

L. M. Dalcher, Secretary of the Committee, is in military service and S. A. Greenberg has been appointed to serve as acting secretary of the committee in Mr. Dalcher's absence.

The following two filler metal specifications which have been in effect for more than three years, but which were extensively revised and reissued in 1942, have been placed under the jurisdiction of the joint committee:

Tentative Specifications for:

Iron and Steel Gas-Welding Rods (A 251 – 42 T), and

Iron and Steel Arc-Welding Electrodes (A 233 – 42 T).[1]

NEW TENTATIVE STANDARD

The committee recommends that the proposed Tentative Specifications for Aluminum and Aluminum-Alloy Metal Arc-Welding Electrodes[2] be accepted for publication as tentative.

This recommendation has been submitted to letter ballot of the committee which consists of 68 members; 52 members returned their ballots, of whom 35 have voted affirmatively, 0 negatively, and 17 members marked their ballots "not voting."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Aluminum and Aluminum Alloy Filler Metal (G. O. Hoglund, chairman) has prepared the Specifications for Aluminum and Aluminum-Alloy Metal Arc-Welding Electrodes[2] and recommends them for publication as tentative.

Subcommittee IV on High-Alloy Steel Filler Metal (R. D. Thomas, chairman) has been working on specifications for stainless steel arc-welding electrodes for welding steels of high hardenability, but it has been decided not to recommend this for publication as A.W.S.-A.S.T.M. specifications. The subcommittee is, however, working on other specifications for stainless steel arc-welding electrodes on which it will report later this year.

Subcommittee V on Nickel and Nickel Alloy Filler Metal (O. B. J. Fraser, chairman) has under consideration the preparation of specifications for nickel and nickel alloy filler metal.

This report has been submitted to letter ballot of the committee which consists of 68 members; 37 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. H. DEPPELER,
Chairman.

L. M. DALCHER,
Secretary.
S. A. GREENBERG,
Acting Secretary.

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[1] See Editorial Note, p. 128.

[2] These specifications were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 260.

EDITORIAL NOTE

Subsequent to the Annual Meeting, the Joint AWS-ASTM Committee on Filler Metal presented to the Society through Committee E-10 on Standards a proposed revision of the Tentative Specifications for Iron and Steel Arc-Welding Electrodes (A 233 – 42 T). The revised specifications were accepted by Committee E-10 on October 7, 1943, and assigned the designation A 233 – 43 T. They appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 157.

REPORT OF COMMITTEE B-1

ON

COPPER AND COPPER-ALLOY WIRES FOR ELECTRICAL CONDUCTORS

Committee B-1 on Copper and Copper-Alloy Wires for Electrical Conductors held no meetings during the year.

The committee lost an active member in the death of Stanton Hertz of the Copperweld Steel Co.

There were several changes in membership during the year, as follows:

Secretary H. H. Stout, Jr., is now a Major in the Ordnance Dept., Frankford Arsenal, Philadelphia, Pa., and as such constitutes a new general interest member.

B. J. Sirois replaces Major Stout as representative of Phelps Dodge Copper Products Corp.

M. J. Donachie replaces Paul Beck as one of the representatives of the Beryllium Corp. of Pennsylvania.

A. L. Malmstrom represents the Detroit Edison Co., replacing W. C. Gray.

C. T. Hatcher represents the Consolidated Edison Co. of N. Y., Inc., a new consumer member.

Rolf Selquist represents the Copperweld Steel Co., replacing Stanton Hertz.

Charles Ange represents the Okonite Co., replacing C. P. Brodhun, Jr.

Subsequent to the 1942 annual meeting, Committee B-1 recommended to Committee E-10 on Standards that Emergency Provisions EA - B 158 be withdrawn inasmuch as the Tentative Specifications for Rope-Lay-Stranded and Bunch-Stranded Copper Cables for Electrical Conductors (B 158 - 41 T) to which they apply had been withdrawn by action at the annual meeting. This recommendation was accepted by Committee E-10 on August 24, 1942.

On December 11, 1942, Emergency Alternate Provisions in the Standard Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, and Soft (B 8 - 41), submitted by Committee B-1, were accepted by Committee E-10 and issued under the designation EA - B 8. They were published in ASTM BULLETIN, No. 120, January, 1943, and have been issued in the form of a sticker for attachment to the standard.

No new standards or revisions of standards have been issued during the year, and it is recommended that the standards and tentative standards under the jurisdiction of the committee be retained in their present status for another year.

No provisions were included in the Emergency Specifications for Lead-Coated and Lead-Alloy-Coated Copper Wire for Electrical Purposes (ES - 1a) issued last year, for test of ductility of coating, as sufficient data were not available at that time. Subcommittee X, under the chairmanship of C. S. Gordon, has carried on correspondence, discussion, and tests in regard to the subject. It is felt that there is not yet sufficient background for recommending a test for inclusion in the emergency specifications, although visual inspection of the coating after bending around a mandrel is being carried out by some.

C. J. Snyder, a member of Subcommittee X, is presenting a paper at the annual meeting entitled "Lead-Alloy-Coated Copper Wire for Electrical Conductors."[1]

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[1] See p. 571.

Committee B-1 represented by Messrs. Alfred Bellis and J. R. Becker are collaborating with Subcommittee V of Committee B-5 on proposed Tentative Specifications for Copper Bus Bars, Tubes, and Shapes with respect to the resistivity requirements of these specifications.

Information has been distributed to members of the committee concerning the proposed simplification of sizes and strandings of wires and cables which is being considered by the War Production Board. The attention of the War Production Board has been called to the simplification already provided for through the application of the following A.S.T.M. specifications for stranded conductors prepared by Committee B-1 in 1942 and previous years:

Standard Specifications for:

Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 – 41),

Tentative Specifications for:

Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members for Electrical Conductors (B 172 – 42 T),

Rope-Lay-Stranded Copper Conductors Having Concentric-Stranded Members for Electrical Conductors (B 173 – 42 T), and

Bunch-Stranded Copper Conductors for Electrical Conductors (B 174 – 42 T).

The use of these A.S.T.M. specifications by purchasers of stranded conductors will, it is felt, accomplish much toward the elimination of special varieties of stranded conductors with consequent increase in productive capacity.

Committee B-1 has registered objection to the promulgation by A.S.A. Sectional Committee B32 on Wire and Sheet Metal Gages of a new wire gage closely approximating the long standing American Wire Gage if the inclusion of copper and copper-alloy wires in this "Proposed American Standard of Preferred Sizes for Bare and Metallic-Coated Round Wires" is contemplated. The suitability and universal use of the American Wire Gage for copper wire makes a change in gages extremely undesirable.

This report has been submitted to letter ballot of the committee which consists of 44 members; 43 members returned their ballots, of whom 41 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. H. FOOTE,
Chairman.

H. H. STOUT, JR.
Secretary.

REPORT OF COMMITTEE B-2
ON
NON-FERROUS METALS AND ALLOYS

Committee B-2 on Non-Ferrous Metals and Alloys held one meeting, on June 24, 1942, in Atlantic City, N. J., during the 1942 Annual Meeting of the Society.

The Advisory Committee also met, and approved numerous changes in membership. On invitation it appointed O. B. J. Fraser to serve on the Joint Committee on Filler Metal, functioning under the joint sponsorship of the American Welding Society and the American Society for Testing Materials, which is responsible for the development of specifications for all types of welding rods and electrodes.

The Advisory Committee also received as a matter of information a tentative classification of commercial coppers prepared by a special group of Committee B-5 on Copper and Copper Alloys and voted that efforts to draw up specifications should be confined to specific items on the list, following definite recommendations of a conference to be held by members of this special group of Committee B-5 and the members of Subcommittee I of Committee B-2.

The following standards under the jurisdiction of the committee have been approved as American Standard by the American Standards Association and assigned the ASA numbers indicated:

A.S.T.M. Standard Specifications for:

Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 - 42; ASA H17.1—1942),

Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 - 42; H17.2—1942),

Slab Zinc (Spelter) (B 6 - 37; H24.1—1943), and

Rolled Zinc (B 69 - 39; H25.1—1943).

Subsequent to the annual meeting, Commttee B-2 recommended changes in the Emergency Alternate Provisions EA - B 23 in the Standard Specifications for Babbitt Metal (B 23 - 26) and in EA - B 32 in the Tentative Specifications for Soft Solder Metal (B 32 - 40 T), referred to later in this report, which were accepted by Committee E-10 on Standards on January 5, 1943. The revised emergency alternate provisions EA - B 23a and EA - B 32a were published in ASTM BULLETIN, No. 120, January, 1943, and have also been issued in the form of stickers for attachment to the standards to which they apply.

I. REVISION OF TENTATIVE STANDARD

The committee recommends that the Tentative Specifications for Oxygen-Free Electrolytic Copper Wire Bars, Billets, and Cakes (B 170 - 42 T)[1] be revised, as referred to in the report of Subcommittee I, and continued as tentative.

II. REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 - 42),[1] as referred to in the report of Subcommittee I, and accordingly asks for a nine-tenths affirmative vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society.

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[1] 1942 Book of A.S.T.M. Standards, Part I.

III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative standards be approved for reference to letter ballot of the Society for adoption as standard, with revisions in the Specifications B115, as referred to in the reports of the subcommittees:

Tentative Specifications for:[1]

Electrolytic Cathode Copper (B 115 - 41 T), as proposed by Subcommittee I,
Pig Lead (B 29 - 40 T), proposed by Subcommittee II,
Phosphor Copper (B 52 - 38 T), proposed by Subcommittee VIII,
Silicon Copper (B 53 - 38 T), proposed by Subcommittee VIII, and
Brazing Solder (B 64 - 38 T), proposed by Subcommittee VIII.

These recommendations have been submitted to letter ballot of the committee which consists of 115 voting members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVE STANDARD			
Spec. for Oxygen-Free Electrolytic Copper Wire Bars, Billets, and Cakes (B 170 - 42 T)	47	0	12
II. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Spec. for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 - 42)	47	0	12
III. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Spec. for Electrolytic Cathode Copper (B 115 - 41 T)	46	0	13
Spec. for Pig Lead (B 29 - 40 T)	65	1	18
Spec. for Phosphor Copper (B 52 - 38 T)	44	0	15
Spec. for Silicon Copper (B 53 - 38 T)	42	0	17
Spec. for Brazing Solder (B 64 - 38 T)	41	1	17

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Refined Copper (J. L. Christie, chairman) held two meetings, the first on June 23, 1942, in Atlantic City, N. J., but no quorum was present, and the second on May 27, 1943, in New York, N. Y.

At the June meeting one member questioned the proper interpretation of the figures of resistivity given to five decimal places in the specifications for copper for electrical purposes. Action was taken to recommend to the Advisory Committee of Committee B-2 that a footnote to read, "0.15328 internal ohm (meter, gram) is regarded as an expression synonymous with exactly 100 per cent conductivity, and is a matter of definition, and the five places of decimals quoted above, or given as other permissible values of resistivity, do not imply that limits of accuracy to the fifth significant figures must be achieved in the determination of acceptance tests" be added to the following specifications:

Standard Specifications for:

Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 - 42),
Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 - 42),
Electrolytic Cathode Copper (B 115 - 41 T), and
Oxygen-Free Electrolytic Copper Wire Bars, Billets, and Cakes (B 170 - 42 T).

This recommendation was accepted by the Advisory Committee. However, cogent objections were raised by Committee B-1 on Copper and Copper Alloy Wire for Electrical Conductors, and at the May meeting of Subcommittee I, it was voted to rescind such action.

As a result of action taken at the May meeting, the subcommittee also recommends that Tentative Specifications B 115 - 41 T be adopted as standard.

At the June meeting, A. J. Phillips and S. Skowronski were appointed to confer with a group from Committee B-5 on Copper and Copper Alloys, Cast and Wrought, under the chairmanship of H. H. Stout, Jr., on the proposed classification of commercial coppers. At a conference held on October 19, 1942, the following recommendations were unanimously approved:

1. A reference to "silver-bearing copper" should be included in Specifications B 5 and B 170, preferably in the form of a footnote to the effect that silver would be regarded as equivalent to copper in the assay, up to 30 oz. per ton (if agreeable to both the seller and the purchaser).

2. "Casting copper" should be recognized by name in the scope of the Standard Specifications for Fire-Refined Copper Other than Lake (B 72 – 33).

3. A new specification for "phosphorized copper" of high electrical conductivity should be prepared.

4. Subject to approval by the Coordinating and Advisory Committees, a commercial class of metal containing more than 99.90 per cent copper should be regarded as copper metal and hence under the jurisdiction of Subcommittee I of Committee B-2, whereas, metal such as arsenical and phosphor copper containing less than 99.90 per cent copper should be regarded as a copper alloy, and hence under the jurisdiction of Committee B-5.

At the May meeting of Subcommittee I, the following actions were taken with respect to the above recommendations:

1. It was voted that the following note should be added to Specifications B 5 and B 170:

NOTE.—By agreement between the seller and the purchaser, the addition of silver up to 30 oz. per ton will be considered within the specifications; silver being counted as copper in the chemical analysis.

2. The recommendation that casting copper be recognized by name in the scope of Specifications B 72, without further change, was rejected because much so-called casting copper is sold in the form of scrap (without being re-cast into ingots) and will not meet the requirements of these specifications. It was further voted that the development of specifications for lower grade copper should await more definite recommendations from the producers and consumers of this material.

Subcommittee II on Refined Lead, Tin, Antimony, and Bismuth (T. A. Wright, chairman) held a meeting on June 24, 1942, at which the status of the Tentative Specifications for Pig Lead (B 29 – 40 T) was considered at length. Certain former objections had been resolved and withdrawn. It is accordingly recommended that Tentative Specifications B 29 be adopted as standard.

Subcommittee III on White Metal Alloys (G. H. Clamer, chairman), in view of the further restrictions on the use of tin, appointed a group under the chairmanship of G. O. Hiers to revise the Emergency Alternate Provisions for Soft Solder Metal (EA – B 32) and for Babbitt Metal (EA – B 23). This group met on July 29, and unanimously approved changes, principally in the analysis of permissible alloys, which were accepted by Committee E-10 on Standards early in January and issued in the form of stickers under the designations EA – B 32a and EA – B 23a, respectively.

Since radical changes in future commercial practices on dipping or wiping solders may be expected as the result of present restrictions on tin content and experience with the alloys low in tin, it is recommended that the Tentative Specifications for Soft Solder Metal (B 32 – 40 T) be continued as tentative.

Subcommittee VII on Refined Nickel and High Nickel Alloys, Cast and Wrought

(O. B. J. Fraser, chairman), in view of the probability of minor changes in Government requirements, recommends that the following nine specifications be continued as tentative for another year:

Tentative Specifications for:

Nickel Rods and Bars (B 160 – 41 T),
Nickel Cold-Drawn Pipe and Tubing (B 161 – 41 T),
Nickel Plate, Sheet, and Strip (B 162 – 41 T),
Nickel, Nickel-Copper Alloy, and Nickel-Chromium-Iron Alloy Seamless Condenser Tubes and Ferrule Stock (B 163 – 41 T),
Nickel-Copper Alloy Rods and Bars (B 164 – 41 T),
Nickel-Copper Alloy Cold-Drawn Pipe and Tubing (B 165 – 41 T),
Nickel-Chromium-Iron Alloy Rods and Bars (B 166 – 41 T),
Nickel-Chromium-Iron Alloy Cold-Drawn Pipe and Tubing (B 167 – 41 T), and
Nickel-Chromium-Iron Alloy Plate, Sheet, and Strip (B 168 – 41 T).

Subcommittee VIII on Miscellaneous Refined Metals and Alloys (E. E. Schumacher, chairman) recommends that the Tentative Specifications for Phosphor Copper (B 52 – 38 T), for Silicon Copper (B 53 – 38 T), and for Brazing Solder (B 64 – 38 T), which have received no adverse criticism since their revision in 1938, be adopted as standard.

This report has been submitted to letter ballot of the committee which consists of 115 members; 60 members returned their ballots, of whom 54 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. E. THUM,
Chairman.

C. E. SWARTZ,
Secretary.

REPORT OF COMMITTEE B-3
ON
CORROSION OF NON-FERROUS METALS AND ALLOYS

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held a meeting in Atlantic City, N. J., on June 24, 1942, and in Buffalo, N. Y., on March 2, 1943.

The committee consists of 78 members, of whom 64 are voting members; 29 are classified as producers, 23 as consumers, and 12 as general interest members.

NEW TENTATIVE STANDARD

The committee recommends that the new proposed Method of Total Immersion Testing of Non-Ferrous Metals,[1] prepared by Subcommittee I, be accepted for publication as tentative. The recommended method sets forth the minimum requirements for making total immersion tests permitting a wide latitude in the choice of test conditions to cover specific uses of the test.

This recommendation has been submitted to letter ballot of the committee which consists of 64 voting members; 52 members returned their ballots of whom 43 have voted affirmatively, 1 negatively, and 8 members marked their ballots "not voting."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Total Immersion Test (A. W. Tracy, chairman).—Subcommittee I has prepared the method of total immersion testing of non-ferrous metals being recommended for publication as tentative this year.

Subcommittee II on Alternate Immersion Test (H. W. Schmidt, chairman).—A method of alternate immersion testing of non-ferrous metals formulated several years ago will be recirculated among the members of the subcommittee, and from comments received it is expected that a method of alternate immersion testing will be agreed upon for submittal as a tentative standard.

Subcommittee III on Spray Test (C. E. Heussner, chairman).—This subcommittee has been inactive during the past year.

The committee recommends that the Tentative Method of Salt Spray Testing of Non-Ferrous Metals (B 117 - 41 T) be continued as tentative.

Subcommittee V on Statistical Analysis and Planning of Corrosion Testing (W. E. Campbell, chairman).—This subcommittee has supervised the calculation of statistical constants for the data for the 10-yr. period of the atmospheric corrosion tests being carried out by Subcommittee VI.

Subcommittee VI on Atmospheric Corrosion (W. H. Finkeldey, chairman).—A report on the results of tests of 24 non-ferrous metals exposed to atmospheric corrosion for periods of 1, 3, 6, and 10 yr. has been prepared by the subcommittee, as appended hereto.

Subcommittee VII on Weather (F. L. LaQue, chairman).—In accordance with the previously established policy of this subcommittee, no program of action will be put into effect until after the war. In the meantime, the subcommittee is considering plans for such future activity as have been and may be indicated.

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[1] This method was accepted as tentative by the Society and appears in the 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 332.

Subcommittee VIII on Galvanic and Electrolytic Corrosion (L. J. Gorman, chairman).—Atmospheric galvanic corrosion tests on type 304 and type 316 stainless steels, coupled with various other metals, have been in progress at New York, N. Y.; Altoona, Pa.; State College, Pa.; and Wilmington, N. C., since 1941. The mild steel disks are showing rapid corrosion, and a complete analysis of the steel has shown an abnormally low copper content. New couples using mild steel with a normal copper content will be exposed at the several locations. The subcommittee will also expose type 430 stainless steel coupled with 24ST aluminum alloy.

This report has been submitted to letter ballot of the committee which consists of 64 voting members; 52 members returned their ballots, of whom 51 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

SAM TOUR,
Chairman.

A. W. TRACY,
Secretary.

REPORT OF SUBCOMMITTEE VI ON ATMOSPHERIC CORROSION OF NON-FERROUS METALS AND ALLOYS

The initial program of Subcommittee VI on Atmospheric Corrosion of Non-Ferrous Metals and Alloys contemplated making five separate tests after various time intervals on the 24 non-ferrous metals and alloys[1] exposed in 1931 to the atmospheres at nine test locations. Three sets of tests have already been made after exposure intervals of approximately 1, 3, and 6 yr.[2] Late in the summer of 1941, the fourth removal of specimens was made, and the specimens were tested in accordance with the procedure followed in the previous tests, that is, determination of the change in weight on the 9 by 12-in. plate specimens as removed from the racks (not chemically cleaned), and the tensile strength and percentage elongations measured on the tension specimens.

When these tests were completed, the subcommittee decided not to return these so-called "removable" plates to the test racks at the various locations for further exposure, as had been done previously, but to clean the plates chemically to remove corrosion products and reweigh them in order to determine the true loss in weight due to corrosion. It was further decided to cut standard tension specimens from the plates and test these specimens for tensile strength and percentage elongation. There is still exposed at seven of the test locations a "permanent" set, consisting of two 9 by 12 in. plates of each of the metals and alloys on which the exposure period has never been interrupted for inspection and weighing. The exposure of these plates will be continued until such time as the committee decides to terminate the tests.

Since the latest complete report of the subcommittee in 1938, certain acts of vandalism have occurred at several of our test locations resulting in the destruction or removal of test specimens. During the spring of 1939, all the plate and tension test specimens were stolen from the racks at the Pittsburgh location (Brunot Island). In the spring of 1941, 9 by 12-in. plate specimens of the lead materials "U" and "CC," and the tin samples "EE" were stolen from the racks at Sandy Hook. Also in the summer of 1941, both sets of plate specimens were stolen from the racks at the Rochester location. In the summer of 1942, for some unexplained reason, the entire test rack was dismantled and scrapped beyond any chance of recovery along with all the specimen racks and the remaining last set of tension specimens, by some employees of the Rochester Gas and Electric Co., on whose property the test rack was located. Fortunately, the removal of the 10-yr. exposure tension test specimens was made before this last mishap occurred.

Although our test materials are protected at all ground locations with standard chain-link wire fencing enclosures topped with double strands of barbed wire, such measures are not sufficient protection against vandalism unless the test site is located where it is under constant supervision of responsible individuals.

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[1] A full description of the metals and alloys included in these tests giving chemical composition, mechanical properties, and microstructure appears in the 1932 Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 214 (1932).

[2] The results of previous tests, made after 1-yr., 3-yr., and 6-yr. exposures are given in the respective Reports of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 234 (1933); Vol. 35, Part I, p. 142 (1935); and Vol. 38, Part I, p. 194 (1938).

In the fall of 1940, it was necessary to move the entire test rack and enclosure fence at Key West to a new location in the same area owing to the use of the former test site by the U. S. Navy. The new location is fairly close to the first test site, and it is believed that it will differ little in its corrosion characteristics from the former location.

TABLE I.—LENGTH OF EXPOSURE PERIODS AT VARIOUS TEST LOCATIONS.

Location	9 by 12-in. Plate Specimens										Tension Specimens							
	First Period		Second Period		Third Period		Fourth Period		Total Exposure Time		First Period		Second Period		Third Period		Fourth Period	
	Days	Years	Days	Years	Days	Years	Days	Years	Days	Years	Days	Years	Days	Years	Days	Years	Days	Years
No. 1, Pittsburgh, Pa.	428	1.17	622	1.71	984	2.70	Missing		2034	5.58	428	1.17	1154	3.16	2232	6.11	Missing	
No. 2, Altoona, Pa.	438	1.20	607	1.66	973	2.67	1359	3.72	3377	9.25	438	1.20	1148	3.14	2219	6.07	3695	10.11
No. 3, State College, Pa.	422	1.15	642	1.76	972	2.66	1350	3.70	3386	9.27	422	1.15	1170	3.21	2217	6.07	3687	10.09
No. 4, New York, N. Y.	456	1.25	627	1.72	972	2.66	1371	3.75	3426	9.38	456	1.25	1205	3.30	2256	6.18	3741	10.24
No. 5, Sandy Hook, N. J.	470	1.29	626	1.72	988	2.71	1365	3.73	3449	9.45	470	1.29	1198	3.28	2273	6.22	3733	10.22
No. 6, Rochester, N. Y.	265	0.73	615	1.69	971	2.66	Missing		1851	5.08	420	1.15	1145	3.14	2215	6.06	3694	10.11
No. 7, Key West, Fla.	416	1.14	632	1.73	988	2.71	1366	3.74	3402	9.32	416	1.14	1145	3.14	2227	6.10	3693	10.11
No. 8, Phoenix, Ariz.	389	1.06	623	1.71	942	2.58	1369	3.75	3323	9.10	389	1.06	1111	3.04	2174	5.95	3650	10.00
No. 9, La Jolla, Calif.	380	1.04	614	1.68	960	2.63	1382	3.79	3336	9.14	380	1.04	1101	3.02	2164	5.92	3636	9.95

Exposure Periods:

The length of the exposure periods at each test location for the 9 by 12-in. plate specimens and for the tension specimens is given in Table I. It should be remembered that the total exposure time for the plate specimens is the sum of the actual exposure periods, and does not, of course, include the time when the specimens were off the racks for weighing and inspection at the time of the 1-, 3- and 6-yr. exposure tests. This explains the difference in exposure periods of the plate specimens and the tension specimens, whose exposure time was continuous.

Change in Weight Tests:

In accordance with the procedure followed in the previous tests, the 9 by 12 in. plate specimens were weighed in the usual manner by the Division of Metallurgy of the National Bureau of Standards, just as they came from the exposure racks, without attempting to remove any accumulation of corrosion products. The weight determinations were made on a special balance, sensitive to 0.1 mg.; however, the weights of the plate specimens are reported only to 0.01 g. After the first weighing, the plates were given the usual visual inspection by the committee as a whole. The results of this inspection are given in detail in a later section of this report. The plate specimens were then sent to the laboratories of the various suppliers of the metals and alloys for chemical cleaning to remove all corrosion product films, and were then returned to the Bureau of Standards for reweighing after cleaning. In general, the cleaning methods used for removing corrosion products were the same as those used to remove the corrosion products from the electrolytic corrosion couple specimens exposed by Subcommittee VIII on Galvanic and Electrolytic Corrosion and described in detail in its report for 1934.[3]

Since no standard units have as yet been adopted for reporting weight changes, the figures appearing in the tables are the total weight changes in grams for each 9 by 12-in. plate specimen, prefixed by a plus or minus sign to indicate a gain or loss. The weight changes reported can be converted into units in common use by reference to the tabulation of critical data, and the table of densities published in the 1933 report of Subcommittee VI on Atmospheric Corrosion of Non-Ferrous Metals and Alloys.[4]

In addition to the weight changes for the 10-yr. exposure period on specimens uncleaned and cleaned, there has been included for comparison purposes the weight changes on uncleaned specimens for the 1-, 3-, and 6-yr. exposure periods.

The agreement in the 10-yr. exposure weight changes on duplicate specimens is fairly good in both the weighings before chemical cleaning and after chemical cleaning.

Tension Tests:

Determinations of tensile strength and percentage elongation in 2 in. were made on the eight sets of corroded tension test specimens of each material after an exposure of approximately 10 yr. In each case, these tests were made by the same laboratories which had made the previous tension tests on specimens after the 1-, 3-, and 6-yr. exposure periods, and in general the tension test procedure followed was identical with that used previously. However, with respect to the tension specimens cut from the cleaned 9 by 12-in. plates, certain special methods of sampling were observed, as described in the following paragraphs, and the specimens were tested in a previously determined order with respect to their original position in the 9 by 12-in. plate, in order to permit the application of statistical methods of analysis with greater accuracy.

Before cutting out the tension specimens from the corroded and cleaned 9 by 12-in. plates, it was necessary to determine whether the grain direction in the tension specimens exposed on the rack was parallel to or at right angles to the long dimension of the test specimen. Heretofore, in all previous tests, this matter of grain direction was of no consequence, since the change in tensile properties was only being used as a measure of the corrosion which had taken place. However, in order that comparisons could be made between tension tests on uncleaned specimens and tension tests on specimens cut from chemically cleaned plates, and also comparisons made between tests on specimens with corroded edges and specimens with freshly machined cut edges, it was highly important that the grain direction be the same in both groups of specimens.

The matter was further complicated by the fact that, in some instances, the 9 by 12-in. plates were cut originally from strips rolled 12-in. wide, with the grain direction of the plate parallel to the 9-in. dimension, and in other cases from strips rolled 9-in. wide, with the grain direction of the plate parallel to the 12-in. dimension.

The chemically cleaned plates, after weighing at the Bureau of Standards, were returned to the laboratories of the companies assigned to test these materials, and were there cut up into tension test specimens whose long axis was either

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[3] Report of Subcommittee VIII, of Committee B-3, on Galvanic and Electrolytic Corrosion, *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 236 (1934).

[4] Report of Subcommittee VI, of Committee B-3, on Atmospheric Corrosion of Non-Ferrous Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 237 (1933).

TABLE II.—SUMMARY OF METEOROLOGICAL DATA AT TEST LOCATIONS.

Station		No. 1 Pittsburgh, Pa.	No. 2 Altoona, Pa.	No. 4 New York, N. Y.	No. 6 Rochester, N. Y.	No. 5 Sandy Hook, N. J.	No. 7 Key West, Fla.	No. 9 La Jolla, Calif.	No. 3 State College, Pa.	No. 8 Phoenix, Ariz.
Type of Exposure		Industrial	Indus.	Industrial	Industrial	Seacoast	Seacoast	Seacoast	Rural	Rural
Elevation, ft.		842	1615	150	523	22	22	50	1217	1108
Temperature, Mean Annual, deg. Fahr.	1931	55	....	55	51	55	76	64	52	72
	1932	53.5	50	54	49	54	78	61	50	71
	1933	53.6	50	54	50	....	78	60	51	71
	1934	52.7	....	52.2	47.9	52	77.5	63.3	49.7	74
	1935	52.1	....	52.6	47.8	52.4	77.2	61.5	49.1	71
	1936	51.0	48.6	52.9	48.2	53	77.4	62.8	49.0	73
	1937	50.9	48.7	53.9	48.8	53.9	77.4	61.5	49.3	71
	1938	53.0	50.2	54.4	50.2	54	77.7	62.2	50.6	71
	1939	52.8	....	53.7	48.8	53.7	77.9	62.9	50.7	71
	1940	49.6	....	51.2	46.4	51.2	76.2	63.6	47.4	73
	1941	52.3	50.0	54.2	48.3	54.2	77.3	64.1	50.4	70
Precipitation, Yearly Total, in.	1931	39	....	35	31	32	51	15	34	10
	1932	25.9	38	39	40	36	40	11	38	7
	1933	40.3	51	50	26	....	52	8	44	7
	1934	31.67	36.41	44.96	24.46	41.00	31.50	8.87	35.54	5.87
	1935	34.44	37.71	32.64	29.65	32.16	39.62	10.20	37.41	10.33
	1936	38.47	47.31	46.33	30.08	42.68	50.46	14.42	47.12	8.29
	1937	37.66	49.20	48.1	33.03	41.47	49.3	10.09	45.67	5.37
	1938	33.2	30.72	46.40	29.10	50.95	22.58	13.01	33.15	4.74
	1939	31.59	....	35.69	25.19	35.69	42.12	9.97	38.68	9.12
	1940	41.61	....	45.03	28.60	45.03	41.50	14.75	39.77	8.48
	1941		42.99	36.20	24.02	36.20	52.01	24.93	30.26	17.26
Snowfall, in.	1931	16	....	7	63	4	0	0	24	0
	1932	19.5	44	13	79	12	0	0	30	0
	1933	20.6	31	28	57	....	0	0	28	0
	1934	24.2	....	38.8	55.5	29.9	0	0	22.6	0
	1935	41.8	55.6	39.0	63.2	34.1	0	0	53.2	0
	1936	53.3	64.0	25.3	82.8	9.2	0	0	65.4	0
	1937	23.1	34.0	11.3	58.50	8.4	0	0	31.9	0
	1938	23.5	36.0	26.2	56.2	18.6	0	0	27.8	0
	1939	29.7	....	22.1	66.3	22.1	0	0	39.4	0
	1940	44.8	....	23.8	72.3	23.8	0	0	46.6	0
	1941	34.7	64.8	30.0	59.3	30.0	0	0	40.5	0
Number of Days Precipitation (rain or snow) 0.01 in. or over	1931	176	....	151	154	116	119	48	113	38
	1932	133	129	116	168	121	119	40	117	37
	1933	140	156	129	160	....	105	40	149	37
	1934	125	....	122	161	129	124	38	117	26
	1935	159	131	120	168	131	100	43	139	47
	1936	142	126	139	165	127	132	54	124	39
	1937	155	130	137	162	128	124	54	140	32
	1938	142	122	139	142	147	100	52	133	29
	1939	155	....	117	150	117	115	50	131	35
	1940	172	....	134	175	134	107	49	131	45
	1941	128	119	94	166	94	107	75	114	63
Relative Humidity, percent	1931	73	....	66	40	73	77	75	....	38
	1932	66	....	63	68	71	74	74	....	34
	1933	68	....	67	66	....	76	75	....	34
	1934	69	....	65	66	73	75	77	....	32
	1935	74	....	66	69	76	73	75	....	38
	1936	71	....	66	68	74	76	75	....	34
	1937	69	....	63	66	75	76	75	....	36
	1938	71	....	65	68	77	76	72	....	24
	1939	71	....	61	74	61	77	72	....	26
	1940	74	....	63	77	63	78	82	....	27
	1941	62	....	58	70	58	79	65	....	34
Sunshine, Percentage of Possible	1931	54	....	62	50	55	66	67	....	84
	1932	58	....	62	48	58	70	65	....	84
	1933	50	....	57	46	....	70	67	....	84
	1934	56	....	60	47	54	71	71	....	85
	1935	50	....	56	47	58	74	62	....	75
	1936	62	....	63	48	62	69	75	....	84
	1937	52	....	59	48	58	67	67	....	84
	1938	56	....	61	50	57	69	68	....	83
	1939	54	....	56	54	56	70	67	....	85
	1940	44	....	57	51	57	68	65	....	79
	1941	50	....	69	56	69	62	66	....	83
Wind, Average Hourly Velocity, mph., and Prevailing Direction	1931	8.7 W.	....	13.4 N.W.	8.7 W.	12.7 W.	9.1 E.	6.6 W.	...	6.0 E.
	1932	10.0 W.	... W.	15.1 N.W.	9.2 W.	14.6 W.	9.7 E.	6.5 W.	... W.	6.1 E.
	1933	10.05 W.	... W.	14.2 N.W.	8.8 W.	....	9.9 E.	6.4 N.W.	... W.	6.0 E.
	1934	9.5 S.W.	... W.	13.8 N.	8.1 S.W.	13.6 S.W.	9.4 E.	6.4 W.	... W.	5.9 E.
	1935	9.8 S.W.	... W.	14.2 N.	8.4 S.W.	13.9 S.W.	9.6 E	6.3 N.W.	... W.	5.7 E.
	1936	10.6 S.W.	... W.	14.5 N.W.	9.0 S.W.	14.1 W.	9.9 E.	6.7 N.W.	... W.	5.8 E.
	1937	10.7 N.W.	... W.	14.0 N.W.	9.3 S.W.-W.	13.9 W.	9.7 E.	6.5 N.W.	... W.	5.9 E.
	1938	10.3 S.W.	...	13.7 N.	8.6 S.W.	13.4 S.	9.8 E.	6.7 N.W.	... W.	5.9 E.
	1939	11.0 S.W.	....	14.4 N.W.	8.9 S.W.	....	9.4 E.	6.5 N.W.	... W.	5.7 E.
	1940	10.4 N.W.	...	13.9 N.W.	8.7 W.	....	9.7 E.	6.7 N.W.	... W.	5.7 E.
	1941	10.4 N.W.	... W.	15.3 N.W.	10.5 S.W.	....	9.3 E.	6.7 N.W.	... W.	5.3 E.

parallel to the 9 or the 12-in. dimension, depending upon the grain direction in the 9 by 12-in. plate, and the grain direction in the tension specimens exposed on the test racks.

The tension tests on these specimens could not be completed in time for inclusion in this report. These data will appear in next year's annual report.

Statistical Analyses of Tension Test Data:

In the previous reports of this subcommittee covering the tests made after 1-, 3-, and 6-yr. exposure periods, the results of a statistical analysis of the tension test data were used to calculate what portion of the change in tensile properties was due to pure chance. A figure expressing this calculation was included in the tabulations under the designation "Maximum Percentage Difference Due to Chance." Following the publication of the 1938 report covering the tests made after 6-yr. exposure, the applicability of the statistical methods used in analyzing the data was the subject of considerable discussion, and a special subgroup was appointed in 1938 to investigate the whole matter of the statistical analysis of the tension data. They recommended the discontinuance of the calculation of the "maximum difference due to chance" because of the inadequacies of the tension test data. The complete findings of this subgroup are included in the report of Subcommittee VI for 1941.[5] Appearing as part of this report are recalculations for each of the materials of the average tensile strength in pounds per square inch, and the standard deviation in pounds per square inch for each of the sets of tension test data covering the 1-, 3-, and 6-yr. exposure periods. A description of the statistical methods and formulas used in making these calculations are given in the above-mentioned subcommittee report.

In accordance with the recommendations made by the subgroup on statistical analyses, only the average tensile strength in pounds per square inch, the percentage change in tensile strength from the original, and the standard deviations in pounds per square inch, have been calculated for the 10-yr. exposure on the tensile specimens exposed on the test rack, as well as the specimens cut from the corroded and cleaned 9 by 12-in. plates. These data are given in the tabulations appended to this report.

VISUAL INSPECTION OF CORRODED SPECIMENS

The 9 by 12-in. plate specimens were inspected at the National Bureau of Standards at a meeting of the subcommittee on February 4, 1942. The appearance of the various materials after approximately 10-yr. exposure at each of the test locations is briefly described under the last column in the tabulations appended to this report. In these descriptions, the terms "tarnish," "film," and "scale" have been used in much the same manner as in the previous inspections. A definition of each of these terms is given in the subcommittee's report for 1938.[6]

Through the courtesy of the Eastman Kodak Co., Kodachrome transparencies were made of a number of the corroded plates selected at the time of the inspection meeting at Washington. These illustrate the color and appearance of corrosion films that were typical of the different metals and alloys in various types of atmospheres.

TEST ATMOSPHERES

The meteorological data from the nine test locations as compiled from the regular official Government Weather Bureau Reports, are given in Table II. However, this information appears to be of little value in the evaluation of the corrosiveness of the atmosphere at the various test locations, a point which has received consideration in previous reports. The discussion of this matter and the classification of test location atmospheres appearing in the 1938 report[6], still cover the subject adequately in the light of the information gained from a study of the 10-yr. exposure test data.

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[5] Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 41, Part I, p. 172 (1941).

[6] Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 194 (1938).

CONCLUSIONS

The subcommittee has decided to postpone any interpretation of the test data until the tension test on the specimens cut from the chemically cleaned 9 by 12-in. plates have been completed. It is planned to include in next year's report conclusions covering the test results to date. The subcommittee will also sponsor a number of papers, to be prepared by members of the committee, discussing the atmospheric corrosion behavior of the various metals and alloys included in these tests.

Respectfully submitted on behalf of the subcommittee,

W. H. FINKELDEY,
Chairman.

CHANGES DUE TO ATMOSPHERIC CORROSION

Location	Original Weight, g., 9 by 12-in. Plate Specimens	Actual Change in Weight, g., 9 by 12 in. Plate Specimens					Tensile Properties After 10-yr. Exposure—Specimens as Exposed on Test Racks				Change in Elongation Before Chemical Cleaning, per cent	Description of Corrosion Product Films on 9 by 12-in. Plate Specimens as Removed from Racks
		After 1 yr.	After 3 yr.	After 6 yr.	After 10 yr. Uncleaned	After 10 yr. Chemically Cleaned	Tensile Strength, psi., Average	Change in Tensile Strength, per cent	Standard Deviation, psi., Average	Elongation, per cent, Average		
Material A—Commercial Copper (Tough Pitch) (99.9+ Cu, 0.026 O)												
Pittsburgh No. 1	530.85 526.79	+1.44 +1.42	+0.07 −0.07	−2.34 −2.48	......	......	......	......	......	......	......	
Altoona No. 2	528.08 532.16	+0.26 +0.29	−0.93 −1.04	−3.02 −2.95	−7.00 −6.77	−14.84 −14.66	31 370	−10.9	222.3	43.3	−2.12	Scale—rough, greenish black, uniform, adherent, sooty.
New York City No. 4	560.96 557.28	−0.83 −0.83	−2.77 −2.81	−5.42 −5.52	−9.15 −9.39	−14.83 −15.12	31 560	−10.3	176.8	44.5	−4.9	Film—smooth, red-brown, greenish-gray, overcast, adherent.
Rochester No. 6	539.54 532.08	+0.30 +0.28	+1.54 +1.49	+2.83 +2.60	......	......	32 130	−8.7	253.0	43.7	−3.06	
Sandy Hook No. 5	562.78 560.59	+0.49 +0.59	+0.43 +0.56	+0.19 +0.20	−0.68 −0.97	−7.67 −8.81	32 510	−7.6	118.0	44.6	−5.18	Film—slightly rough, dark gray, with greenish patches, adherent.
Key West No. 7	559.49 559.08	+0.42 +0.43	+0.56 +0.59	+0.72 +0.76	+0.75 +0.84	−6.15 −6.47	33 070	−6.1	146.2	44.8	−5.66	Film—smooth, reddish purple, green on edges, adherent.
La Jolla No. 9	528.87 531.87	+1.54 +1.48	+1.89 +1.78	+2.38 +2.20	+3.01 +2.81	−17.15 −16.01	30 300	−13.9	349.5	37.0	+12.7	Scale, slightly rough, brown, green overcast, adherent.
State College No. 3	560.33 560.99	+0.33 +0.33	+0.57 +0.56	+0.81 +0.80	+0.99 +0.94	−7.14 −7.31	32 260	−8.4	139.3	45.1	−6.36	Film—smooth, black, green overcast, adherent.
Phoenix No. 8	561.29 556.44	+0.12 +0.14	+0.10 +0.13	+0.18 +0.21	+0.33 +0.32	−1.73 −1.70	33 380	−5.2	167.3	44.7	−5.42	Film—smooth, brownish purple, adherent.
Storage, No. 10	......	......	......	......	......	......	33 660	−4.4	167.2	45.0	−6.13	
Material B—Commercial Copper (Phosphorized) (99.9+ Cu, 0.007 P)												
Pittsburgh No. 1	558.66 559.92	+1.27 +1.15	−0.09 −0.21	−2.35 −2.35	......	......	......	......	......	......	......	
Altoona No. 2	553.42 552.29	+0.06 +0.04	−1.17 −1.16	−3.07 −3.00	−5.81 −6.69	−14.40 −14.37	33 880	−3.6	308.1	41.0	−6.97	Film—greenish black, uniform, adherent, sooty.
New York City No. 4	554.57 554.15	−0.84 −0.85	−2.71 −2.72	−5.25 −5.28	−8.79 −8.77	−14.54 −14.44	34 770	−4.0	828.1	41.0	−6.97	Film—smooth, red-brown with greenish-gray overcast, adherent.
Rochester No. 6	557.40 559.36	+0.27 +0.27	+1.40 +1.37	+2.24 +2.24	......	......	34 200	−2.7	341.6	40.5	−4.65	
Sandy Hook No. 5	552.71 551.59	+0.51 +0.49	+0.39 +0.36	0.00 −0.03	−1.23 −1.27	−8.60 −8.46	33 750	−1.1	178.9	41.0	−5.60	Film—slightly rough, dark gray with patches of green, adherent.
Key West No. 7	559.50 554.67	+0.38 +0.38	+0.53 +0.53	+0.74 +0.75	+0.76 +0.79	−0.50 −0.62	35 480	+0.9	122.3	40.0	−3.35	Film—smooth, reddish purple green on edges. adherent.
La Jolla No. 9	554.90 553.64	+1.32 +1.40	+1.58 +1.65	+1.87 +1.94	+2.18 +2.37	−13.07 −14.86	33 300	−5.3	191.5	37.0	+4.39	Scale—fairly rough, brown overcast with green, adherent.
State College No. 3	549.68 553.67	+0.32 +0.32	+0.54 +0.54	+0.75 +0.74	+0.86 +0.85	−6.65 −6.58	34 920	−0.7	176.5	41.0	−6.97	Film—smooth, with slight pitting, black with green overcast, adherent.
Phoenix No. 8	555.28 554.57	+0.11 +0.13	−0.09 +0.10	+0.18 +0.19	+0.28 +0.29	−1.42 −1.47	35 830	+1.9	125.7	41.5	−7.32	Film—smooth, reddish brown, adherent.
Storage, No. 10							36 020	+2.8	307.4	41.0	−6.97	

CHANGES DUE TO ATMOSPHERIC CORROSION

Location	Original Weight, g., 9 by 12-in. Plate Specimens	Actual Change in Weight, g., 9 by 12-in. Plate Specimens					Tensile Properties After 10-yr. Exposure—Specimens as Exposed on Test Racks				Change in Elongation Before Chemical Cleaning, per cent	Description of Corrosion Product Films on 9 by 12-in. Plate Specimens as Removed from Racks
		After 1 yr.	After 3 yr.	After 6 yr.	After 10 yr. Uncleaned	After 10 yr. Chemically Cleaned	Tensile Strength, psi., Average	Change in Tensile Strength, per cent	Standard Deviation, psi., Average	Elongation, per cent, Average		
Material C—Copper-Silicon-Manganese Alloy (96 Cu, 3 Si, 1 Mn)												
Pittsburgh No. 1	543.63 538.80	+1.42 +1.16	+0.68 +0.47	−0.88 −1.07	....	....	....	....	....	....	....	
Altoona No. 2	538.17 537.16	+0.18 −0.15	−0.63 −1.09	−2.43 −2.88	−5.70 −6.07	−16.96 −15.59	54 000	−8.6	778.8	59.4	−6.22	Scale—slightly rough, greenish black, adherent.
New York City No. 4	534.56 536.72	−0.89 −0.88	−2.63 −2.65	−5.81 −5.78	−9.97 −9.78	−18.77 −18.49	53 860	−8.8	524.5	59.3	−6.46	Film—fairly smooth, brownish red with greenish outer film, adherent.
Rochester No. 6	525.96 543.12	+0.27 +0.26	+1.33 +1.25	+2.40 +2.09	....	....	57 230	−3.1	1534	58.7	−5.38	
Sandy Hook No. 5	534.64 534.88	+0.47 +0.49	+0.75 +0.78	+0.32 +0.53	−2.22 −2.01	−13.69 −13.83	55 390	−6.2	612.3	59.6	−7.0	Film—smooth, light grayish green, adherent.
Key West No. 7	539.43 540.13	+0.44 +0.42	+0.84 +0.78	+1.35 +1.28	+2.09 +1.92	−12.26 −11.81	56 810	−3.8	688.2	58.4	−4.84	Film—smooth, brownish red, adherent.
La Jolla No. 9	543.38 543.23	+1.92 +1.77	+3.06 +2.92	+4.37 +4.33	+5.42 +5.57	−22.45 −21.03	55 510	−6.0	1402	58.1	−4.3	Scale—fairly rough, brown with outer green film, adherent.
State College No. 3	545.71 534.63	+0.11 −0.05	+0.16 −0.10	+0.11 −0.13	+0.26 +0.08	−6.51 −4.87	57 020	−2.7	1009	58.3	−4.66	Film—slightly rough dark gray, spotted green in streaks, adherent.
Phoenix No. 8	542.32 537.15	+0.12 +0.16	+0.09 +0.10	+0.18 +0.20	+0.33 +0.36	−2.11 −2.17	57 570	−2.5	598.3	59.1	−6.10	Film—smooth, brownish-red, slight tinge of green, adherent.
Storage, No. 10	......	......	......	......	......	......	58 490	−1.0	626.3	58.5	−5.02	
Material D—Tin Bronze (92 Cu, 8 Sn)												
Pittsburgh No. 1	541.68 541.63	+2.23 +2.26	+2.90 +2.51	−0.14 −0.53	....	....	....	....	....	....	....	
Altoona No. 2	551.10 551.29	+0.79 +0.83	−0.67 −0.57	−4.44 −4.63	−9.44 −9.84	−17.92 −18.35	54 200	−8.1	604.3	66.8	−0.902	Film—fairly rough, black, adherent, sooty.
New York City No. 4	554.93 549.98	−0.22 −0.19	−2.57 −2.08	−7.71 −7.12	−14.87 −14.74	−22.48 −22.62	53 700	−8.8	533.3	66.3	−0.151	Film—rough, red with mottled gray green overcast, adherent.
Rochester No. 6	540.68 542.83	+0.31 +0.30	+1.61 +1.51	+4.06 +3.98	....	....	56 000	−5.0	710.6	65.3	+1.35	
Sandy Hook No. 5	529.86 537.02	+0.63 +0.65	+0.83 +0.86	+0.95 +0.81	+0.49 −0.03	−15.09 −16.10	55 500	−5.9	882.7	66.5	−0.453	Scale—rough, very dark gray with patches of light green, adherent.
Key West No. 7	547.13 536.40	+0.52 +0.53	+0.89 +0.89	+1.17 +1.12	+1.48 +1.36	−9.54 −9.79	56 200	−4.7	981.1	67.7	−2.26	Scale—smooth, brownish red, adherent.
La Jolla No. 9	543.77 547.70	+1.83 +1.71	+2.37 +2.13	+2.08 +1.70	+1.03 +0.81	−25.74 −25.49	55 000	−8.3	732.1	66.7	−0.755	Scale—fairly smooth, greenish over reddish brown, nonadherent.
State College No. 3	529.64 534.33	+0.11 +0.14	+0.25 +0.31	+0.27 +0.36	+0.22 +0.33	−4.03 −4.19	56 600	−4.0	1058	65.8	+0.604	Film—fairly smooth, dark brownish with gray-green overcast, adherent.
Phoenix No. 8	551.11 533.77	+0.14 +0.12	+0.11 +0.10	+0.22 +0.21	+0.42 +0.41	−1.98 −2.01	58 000	−1.5	1078	67.2	−1.5	Film—slightly rough on one side, yellowish brown with traces of tarnish.
Storage, No. 10	......	......	......	......	......	......	58 400	−0.9	212.4	66.5	−0.453	

CHANGES DUE TO ATMOSPHERIC CORROSION

Location	Original Weight, g., 9 by 12-in. Plate Specimens	Actual Change in Weight, g., 9 by 12-in. Plate Specimens					Tensile Properties After 10-yr. Exposure—Specimens as Exposed on Test Racks				Change in Elongation Before Chemical Cleaning, per cent	Description of Corrosion Product Films on 9 by 12-in. Plate Specimens as Removed from Racks
		After 1 yr.	After 3 yr.	After 6 yr.	After 10 yr. Uncleaned	After 10 yr. Chemically Cleaned	Tensile Strength, psi., Average	Change in Tensile Strength, per cent	Standard Deviation, psi., Average	Elongation, per cent, Average		
Material F—Aluminum Bronze (92 Cu, 8 Al)												
Pittsburgh No. 1	490.85 479.69	+1.57 +1.60	+2.43 +2.28	−0.09 −0.53	....	....	......	......	......	......	....	
Altoona No. 2	501.76 515.14	+0.34 +0.47	+0.59 +0.86	+0.56 +0.85	+0.29 +0.44	−18.31 −19.15	70 300	−5.1	737.1	48.58	−4.24	Scale—rough, black, adherent, sooty.
New York City No. 4	496.21 506.66	−0.03 −0.01	−0.55 −0.47	−1.09 −0.97	−2.90 −3.12	−18.45 −18.90	68 616	−7.4	1491	48.08	−3.17	Scale—rough, bluish black, somewhat mottled with green, adherent.
Rochester No. 6	598.64 492.98	+0.18 +0.20	+1.44 +1.50	+4.44 +4.58	....	....	70 866	−4.3	426.3	46.08	+1.11	
Sandy Hook No. 5	491.98 495.54	+0.31 +0.31	+0.59 +0.59	+1.47 +1.46	−0.23 −0.36	−15.27 −14.90	72 060	−2.7	887.9	47.67	−2.28	Scale—mottled gray and black, adherent.
Key West No. 7	482.78 490.84	+0.26 +0.07	+0.39 +0.41	+0.55 +0.59	+0.69 +0.77	−3.63 −3.58	73 566	−0.7	3150	40.33	−13.4	Film—smooth, reddish brown, gray-green overcast, adherent.
La Jolla No. 9	493.12 493.38	+0.93 +0.95	+0.93 +0.92	+0.43 +0.40	−0.35 −0.24	−2.15 −2.21	83 220	+0.4	1055	37.33	+6.44	Scale—rough, yellowish film with gray-green overcast, nonadherent.
State College No. 3	493.94 506.41	−0.03 −0.03	−0.12 −0.13	−0.22 −0.24	−0.43 −0.49	−2.18 −2.14	74 233	+0.2	1963	47.33	−1.56	Film—fairly smooth, yellowish brown, adherent.
Phoenix No. 8	498.31 502.84	+0.09 +0.08	+0.04 +0.04	+0.07 +0.08	+0.09 +0.11	−0.68 −0.71	83 016	+0.2	891.2	39.08	+2.05	Tarnish—smooth, yellowish with tinge of brown, adherent.
Storage, No. 10	......	......	......	......	......	......	76 030	+2.6	52.24	41.41	−3.78	
Material G—Commercial Aluminum (99 + Al)												
Pittsburgh No. 1	167.49 167.00	+0.25 +0.23	+0.35 +0.32	+0.71 +0.54	....	....	......	......	......	......	....	
Altoona No. 2	166.78 167.86	+0.31 +0.27	+0.68 +0.53	+1.12 +0.93	+1.93 +1.63	−1.66 −2.26	15 735	−4.7	142.4	9.8	+32.8	Film—rough, black, adherent, sooty.
New York City No. 4	166.28 166.40	+0.39 +0.36	+0.55 +0.50	+0.88 +0.82	+0.92 +0.88	−2.95 −3.13	15 860	−3.4	91.15	9.3	+36.3	Film—rough, gray black with traces of initial metallic luster, adherent, sooty.
Rochester No. 6	169.29 169.02	+0.09 +0.10	+0.25 +0.26	+0.50 +0.48	....	....	16 460	+0.3	116.6	10.8	+26.0	
Sandy Hook No. 5	166.81 168.29	+0.28 +0.26	+0.59 +0.50	+0.59 +0.48	+0.62 +0.47	−0.22 −0.21	16 395	−0.1	88.50	10.3	+29.4	Tarnish—streaked and mottled with dark, rough, film, adherent.
Key West No. 7	166.87 169.48	+0.09 +0.14	+0.32 +0.25	+0.54 +0.47	+0.65 +0.61	−0.35 −0.35	16 635	+1.4	226.6	12.6	+13.6	Tarnish with outer gray, nonuniform loose film.
La Jolla No. 9	168.12 170.14	+0.76 +0.76	+1.42 +1.40	+2.58 +2.51	+4.03 +3.89	−2.64 −2.80	14 525	−11.5	520.0	3.8	+73.9	Scale—rough, white, nonuniform, loose.
State College No. 3	166.24 166.78	0.00 +0.01	−0.01 0.00	−0.01 −0.01	−0.03 −0.03	−0.12 −0.12	16 940	+3.2	134.7	11.9	+18.4	Tarnish—smooth, mottled with white specks.
Phoenix No. 8	168.03 169.65	+0.04 +0.04	+0.01 +0.01	+0.04 +0.03	+0.04 +0.04	−0.01 −0.01	16 630	+1.3	292.1	13.2	+9.58	Tarnish—bright, slightly roughened by white specks.
Storage, No. 10	......	......	......	......	......	......	16 920	+3.1	75.50	13.3	+8.90	

CHANGES DUE TO ATMOSPHERIC CORROSION

Location	Original Weight, g., 9 by 12-in. Plate Specimens	Actual Change in Weight, g., 9 by 12-in. Plate Specimens					Tensile Properties After 10-yr. Exposure—Specimens as Exposed on Test Racks				Change in Elongation Before Chemical Cleaning, per cent	Description of Corrosion Product Films on 9 by 12-in. Plate Specimens as Removed from Racks
		After 1 yr.	After 3 yr.	After 6 yr.	After 10 yr. Uncleaned	After 10 yr. Chemically Cleaned	Tensile Strength, psi., Average	Change in Tensile Strength, per cent	Standard Deviation, psi., Average	Elongation, per cent, Average		
Material I—Aluminum-Manganese Alloy (1 per cent Mn)												
Pittsburgh No. 1	169.23 168.17	+0.21 +0.21	+0.09 +0.09	+0.11 +0.21	....	....	......	......	......	......	......	
Altoona No. 2	166.41 167.31	+0.25 +0.23	+0.26 +0.02	+0.01 −0.25	−0.02 −0.38	−6.00 −5.84	18 480	−9.6	114.6	8.0	+14.8	Film—rough, black, adherent, sooty.
New York City No. 4	166.80 167.83	+0.22 +0.24	+0.21 +0.21	+0.22 +0.22	+1.04 +0.08	−3.89 −3.95	19 365	−5.3	125.5	7.7	+18.0	Film—rough, gray-black, adherent, sooty.
Rochester No. 6	170.47 171.00	+0.13 +0.14	+0.25 +0.26	+0.33 +0.34	....	....	20 625	+0.9	113.4	10.3	−9.57	
Sandy Hook No. 5	167.51 166.29	+0.16 +0.24	+0.25 +0.37	+0.18 +0.30	+0.12 +0.27	−0.51 −0.51	20 015	−2.1	56.79	9.4	0	Film—rough, mottled gray, adherent.
Key West No. 7	167.63 168.93	+0.08 +0.09	+0.15 +0.20	+0.28 +0.34	+0.31 +0.36	−0.31 −0.35	20 545	+0.4	167.6	9.9	−5.31	Film—tarnish overlaid by mottled gray film.
La Jolla No. 9	166.49 166.09	+0.58 +0.56	+0.85 +0.78	+1.56 +1.45	+2.55 +2.50	−2.22 −2.18	19 080	−6.7	348.8	4.9	+47.8	Scale—rough, mottled white, somewhat loose.
State College No. 3	166.83 168.27	+0.10 0.00	+0.08 −0.01	+0.06 −0.04	+0.02 −0.07	−0.05 −0.14	20 580	+0.6	122.7	10.8	−14.8	Tarnish—smooth, light gray.
Phoenix No. 8	167.20 166.87	+0.03 +0.03	+0.01 +0.01	+0.03 +0.04	+0.03 +0.04	−0.01 −0.01	20 835	+1.9	237.9	10.8	−14.8	Tarnish—smooth, gray.
Storage, No. 10	......	......	......	......	......	......	20 635	+0.9	62.92	11.4	−21.2	
Material K—Manganese Bronze (High-Tensile-Strength Yellow Brass) (66.4 Cu, 19.3 Zn, 5.88 Al, 4.6 Fe, 3.78 Mn)												
Pittsburgh No. 1	441.92 474.27	+1.24 +1.29	+2.59 +2.64	+3.35 +1.98	....	....	......	......	......	......	......	
Altoona No. 2	442.70 432.72	+1.16 +1.09	+2.47 +2.31	+4.40 +4.25	+5.21 +7.02	−66.39 −73.75	55 915	−54.2	5 197	1.6	+81.1	Scale—rough, mottled, brown-black, adherent sooty.
New York City No. 4	477.86 489.40	+1.14 +1.12	+1.97 +1.95	+3.16 +3.08	−1.82 −0.29	−60.68 −57.78	56 515	−53.7	4 476	2.2	+74.1	Scale—rough, mottled brownish, adherent, sooty.
Rochester No. 6	459.04 454.23	+0.46 +0.43	+1.88 +1.75	+4.22 +4.19	....	....	83 575	−31.5	16 030	2.5	+70.5	
Sandy Hook No. 5	433.18 499.31	+0.85 +0.73	+1.68 +1.40	+2.85 +2.44	+2.96 +2.98	−34.62 −25.39	97 680	−19.9	3 616	3.6	+57.6	Scale—rough, brown-black, slightly mottled, adherent.
Key West No. 7	503.91 493.35	+0.56 +0.56	+1.74 +1.68	+3.15 +2.87	+3.30 +3.35	−16.16 −15.16	105 030	−13.9	10 720	3.75	+55.8	Scale—rough and grainy, black over dark brown, adherent.
La Jolla No. 9	488.56 465.51	+1.50 +1.65	+2.50 +2.85	+3.90 +5.11	+6.03 +9.00	−13.17 −21.26	92 800	−23.9	8 159	1.5	+82.3	Scale—rough and grainy, brownish black, slightly mottled, adherent.
State College No. 3	449.61 460.53	−0.05 −0.02	−0.17 −0.11	−0.33 −0.24	−0.57 −0.44	−3.51 −3.67	123 630	+1.3	10 910	5.9	+30.5	Film—fairly smooth, dark brown black, adherent.
Phoenix No. 8	463.03 477.77	+0.09 +0.10	+0.07 +0.07	+0.15 +0.16	+0.25 +0.24	−1.21 −1.38	117 150	−4.0	5 483	4.9	+42.3	Film—fairly smooth, yellowish tan, adherent.
Storage, No. 10	......	......	......	......	......	......	121 130	−0.7	3 992	4.25	+50.0	

CHANGES DUE TO ATMOSPHERIC CORROSION

Location	Original Weight, g., 9 by 12-in. Plate Specimens	Actual Change in Weight, g., 9 by 12-in. Plate Specimens					Tensile Properties After 10-yr. Exposure—Specimens as Exposed on Test Racks				Change in Elongation Before Chemical Cleaning, per cent	Description of Corrosion Product Films on 9 by 12-in. Plate Specimens as Removed from Racks
		After 1 yr.	After 3 yr.	After 6 yr.	After 10 yr. Uncleaned	After 10 yr. Chemically Cleaned	Tensile Strength, psi., Average	Change in Tensile Strength, per cent	Standard Deviation, psi., Average	Elongation, per cent, Average		
Material L—Brass (85 Cu, 15 Zn)												
Pittsburgh No. 1	544.98 545.76	+1.36 +1.21	+1.18 +1.19	−0.37 −0.39	......	......	......	......	......	......	......	
Altoona No. 2	546.77 546.45	+0.78 +0.72	+0.54 +0.46	+0.48 −0.99	−5.39 −5.42	−27.44 −26.46	37 225	−6.7	499.8	40.83	−0.31	Scale—fairly rough, black gray with edges of green, adherent.
New York City No. 4	546.73 545.76	−0.07 −0.12	−1.79 −1.72	−6.01 −5.60	−14.41 −12.94	−27.09 −27.73	38 150	−4.4	371.9	40.33	+0.90	Scale—slightly rough, dark gray black overcast with green, adherent.
Rochester No. 6	546.28 546.76	+0.24 +0.27	+1.38 +1.20	+3.46 +3.25	......	......	38 910	−5.8	297.5	38.9	+4.42	
Sandy Hook No. 5	548.18 549.19	+0.37 +0.37	+0.24 +0.23	0.05 0.04	−2.06 −1.76	−14.91 −14.46	39 410	−1.2	417.1	42.5	−4.42	Scale—fairly smooth, dark gray, overcast with grayish green film, adherent.
Key West No. 7	547.89 547.47	+0.24 +0.22	+0.27 +0.25	+0.27 +0.25	+1.15 +0.20	−5.88 −5.75	40 725	+2.0	336.3	40.75	−0.12	Film—smooth, brick red adherent.
La Jolla No. 9	546.90 544.94	+1.15 +1.08	+1.39 +1.31	+1.58 +1.52	+1.73 +1.71	−7.38 −7.38	39 866	−0.1	600.9	41.83	−2.77	Scale—fairly smooth, red brown, overcast with green film, adherent.
State College No. 3	546.82 547.40	+0.01 +0.01	−0.12 −0.01	−0.42 −0.39	−0.83 −0.79	−9.10 −9.35	39 760	−0.4	186.5	40.67	+0.07	Film—fairly smooth, with black overcast of green, adherent.
Phoenix No. 8	543.16 543.81	+0.10 +0.10	+0.07 +0.07	+0.13 +0.11	+0.10 +0.08	−0.77 −1.74	41 208	−3.2	501.5	41.25	−1.35	Film—smooth, reddish brown, adherent.
Storage, No. 10	......	......	......	......	......	......	41 300	+3.4	413.3	40.5	+0.49	
Material M—Brass (70 Cu, 30 Zn)												
Pittsburgh No. 1	535.30 549.14	−0.45 −0.47	−1.86 −2.71	−6.02 −5.99	......	......	......	......	......	......	......	
Altoona No. 2	534.26 562.14	−0.53 −0.50	−2.27 −2.33	−4.95 −4.95	−8.60 −8.77	−30.99 −29.84	33 800	−29.6	675.2	39.0	+2.54	Scale—fairly rough, black and yellow brown, adherent.
New York City No. 4	562.49 559.08	−1.42 −1.47	−4.15 −4.29	−7.80 −7.80	−13.06 −12.64	−26.29 −26.42	35 000	−26.8	118.0	40.0	+2.35	Scale—fairly smooth, iridescent, blue steel gray, adherent.
Rochester No. 6	535.53 534.82	+0.20 +0.19	+0.52 +0.49	+1.36 +1.35	......	......	45 500	−5.4	639.2	52.0	+0.57	
Sandy Hook No. 5	534.17 558.64	+0.21 +0.23	−0.06 −0.10	−0.45 −0.53	−1.88 −1.98	−11.73 −12.22	43 100	−10.4	182.6	47.0	+10.13	Scale—smooth, black overcast with greenish gray, adherent.
Key West No. 7	555.26 532.56	−0.14 +0.16	−0.25 +0.06	−0.38 −0.08	−0.65 −0.36	−4.51 −4.29	48 000	−0.2	772.4	54.5	−4.2	Film—smooth, brick red with outer tinge of green, adherent.
La Jolla No. 9	558.86 534.68	+0.82 +0.89	+0.85 +0.88	+0.82 +0.82	+0.64 +0.62	−3.55 −3.45	45 100	−6.3	508.5	47.5	+9.17	Scale—fairly smooth, gray green, adherent.
State College No. 3	553.82 558.59	−0.05 −0.06	−0.23 −0.24	−0.51 −0.53	−0.91 −0.92	−4.73 −4.76	47 400	−1.4	737.8	55.5	−6.11	Film—smooth, black and gray green, adherent.
Phoenix No. 8	555.43 534.02	+0.12 +0.10	+0.07 +0.06	+0.08 +0.07	−0.04 −0.06	−1.37 −1.35	48 900	+1.6	426.5	57.0	−8.98	Film—smooth, dark red brown, adherent.
Storage, No. 10	......	......	......	......	......	......	49 000	+1.8	467.5	56.5	−8.03	

CHANGES DUE TO ATMOSPHERIC CORROSION

Location	Original Weight, g., 9 by 12-in. Plate Specimens	Actual Change in Weight, g., 9 by 12-in. Plate Specimens					Tensile Properties After 10-yr. Exposure—Specimens as Exposed on Test Racks				Change in Elongation Before Chemical Cleaning, per cent	Description of Corrosion Product Films on 9 by 12-in. Plate Specimens as Removed from Racks
		After 1 yr.	After 3 yr.	After 6 yr.	After 10 yr. Uncleaned	After 10 yr. Chemically Cleaned	Tensile Strength, psi., Average	Change in Tensile Strength, per cent	Standard Deviation, psi., Average	Elongation, per cent, Average		
Material N—Admiralty Metal (70 Cu, 29 Zn, 1 Sn)												
Pittsburgh No. 1	533.72 533.85	+0.69 +0.74	+0.60 −0.28	−5.19 −5.30	....	....	....	....	....	....	......	
Altoona No. 2	533.97 533.14	−0.50 −0.53	−1.45 −1.45	−3.07 −3.08	−6.17 −6.16	−19.58 −19.78	49 500	−10.0	376.7	52	−0.017	Scale—somewhat rough, iridescent, steel gray, adherent, sooty.
New York City No. 4	536.03 535.71	−1.25 −1.26	−3.39 −3.44	−7.12 −7.05	−16.49 −16.01	−23.28 −22.93	50 100	−8.9	581.7	52.8	−0.003	Film—smooth, gray overcast with green, adherent.
Rochester No. 6	531.53 533.94	+0.15 +0.19	+0.71 +0.81	+1.55 +1.73	....	....	51 400	−6.4	411.4	50.8	+0.005	
Sandy Hook No. 5	534.10 532.12	+0.26 +0.24	+0.03 −0.01	−0.59 −0.66	−3.72 −3.82	−13.77 −13.56	51 500	−6.3	191.5	52	−0.017	Film—slightly rough, gray overcast with green, adherent.
Key West No. 7	532.05 535.19	+0.21 +0.22	+0.16 +0.17	+0.10 +0.10	−0.10 −0.09	−4.42 −4.36	53 300	−3.0	176.2	51.7	−0.011	Film—smooth, reddish brown overcast, with tinge of green, adherent.
La Jolla No. 9	533.95 534.27	+0.92 +0.94	+1.26 +1.32	+1.59 +1.67	+1.71 +1.85	−4.62 −4.76	53 000	−3.6	326.6	52	−0.017	Film—fairly smooth, gray with green overcast, adherent.
State College No. 3	533.11 538.25	−0.07 −0.07	−0.27 −0.26	−0.55 −0.55	−0.97 −0.98	−4.75 −4.86	53 200	−3.3	364.0	51.8	−0.13	Film—smooth, black and gray and green, adherent.
Phoenix No. 8	534.10 533.14	+0.09 +0.09	+0.04 +0.04	+0.07 +0.07	−0.04 −0.03	−1.19 −1.20	54 100	−1.7	450.0	50.7	+0.007	Film—smooth, red-brown, adherent.
Storage, No. 10	......	......	......	......	......	......	54 400	−1.1	423.3	50	+0.021	
Material O—Duralumin (4 Cu, 0.5 Mn, 0.5 Mg)												
Pittsburgh No. 1	166.17 167.37	+0.73 +0.84	+0.87 +0.96	+1.22 +1.19	....	....	....	....	....	....	......	
Altoona No. 2	168.35 171.77	+0.68 +0.66	+0.96 +0.94	+1.25 +1.28	+1.68 +1.73	−5.06 −5.45	55 360	−13.2	306.0	9.9	+0.52	Film—fairly smooth, black, adherent, sooty.
New York City No. 4	165.66 164.70	+0.87 +1.04	+1.00 +1.15	+1.19 +1.29	+1.20 +1.32	−4.37 −4.69	56 450	−11.5	433.4	10.4	+0.419	Film—rough, black dotted with red specks, adherent.
Rochester No. 6	168.41 167.83	+0.42 +0.49	+1.33 +1.39	+1.68 +1.75	....	....	60 935	−4.4	763.9	14.0	+0.32	
Sandy Hook No. 5	168.42 172.11	+1.05 +1.09	+1.41 +1.40	+1.44 +1.45	+1.49 +1.54	−0.30 −0.30	58 475	−8.3	270.1	11.4	+0.44	Film—rough, dark gray, slightly mottled with white specks, adherent.
Key West No. 7	171.99 169.38	+0.87 +0.64	+1.17 +0.91	+1.49 +1.15	+1.75 +1.36	−0.69 −0.59	62 290	−2.3	810.2	12.6	+0.39	Film—fairly smooth, gray with white spots of scale, adherent.
La Jolla No. 9	168.13 169.30	+2.89 +2.84	+3.48 +3.42	+7.17 +5.33	+10.09 +9.05	−6.16 −5.26	32 500	−49.0	225.0	1.6	+0.97	Scale—rough, white mottled with gray, nonadherent.
State College No. 3	165.99 165.00	−0.01 −0.01	−0.03 −0.03	−0.04 −0.05	−0.09 −0.08	−0.21 −0.20	65 020	+3.0	109.0	18.9	+0.088	Tarnish—smooth, gray.
Phoenix No. 8	167.44 169.81	+0.06 +0.10	+0.03 +0.07	+0.09 +0.13	+0.11 +0.26	−0.04 −0.03	65 560	+2.7	634.9	17.6	+0.15	Tarnish—smooth, gray.
Storage, No. 10	......	......	......	......	......	......	65 705	+3.0	542.5	20.6	+0.0048	

CHANGES DUE TO ATMOSPHERIC CORROSION

Location	Original Weight, g., 9 by 12-in. Plate Specimens	Actual Change in Weight, g., 9 by 12-in. Plate Specimens					Tensile Properties After 10-yr. Exposure—Specimens as Exposed on Test Racks				Change in Elongation Before Chemical Cleaning, per cent	Description of Corrosion Product Films on 9 by 12- in. Plate Specimens as Removed from Racks
		After 1 yr.	After 3 yr.	After 6 yr.	After 10 yr. Uncleaned	After 10 yr. Chemically Cleaned	Tensile Strength, psi., Average	Change in Tensile Strength, per cent	Standard Deviation, psi., Average	Elongation, per cent, Average		
Material P—Copper-Nickel-Zinc Alloy (75 Cu, 20 Ni, 5 Zn)												
Pittsburgh No. 1	562.71 566.41	+0.25 +0.23	−2.89 −2.52	−6.51 −6.10	....	....	......	......	......	......	......	
Altoona No. 2	571.48 570.17	−0.43 −0.43	−3.52 −3.12	−8.88 −8.12	−17.54 −16.30	−26.85 −27.04	48 130	−11.4	446.4	31.1	+0.012	Scale—fairly rough, black over brown, somewhat loose.
New York City No. 4	570.23 569.48	−1.29 −1.34	−3.95 −4.03	−8.77 −9.16	−15.33 −14.58	−22.49 −22.96	48 850	−10.1	340.6	31.7	−0.006	Scale—fairly smooth, green over gray, adherent.
Rochester No. 6	557.41 556.14	+0.20 +0.19	+0.71 +0.67	+0.72 +0.51	....	....	50 300	−7.4	436.6	30.8	+0.022	
Sandy Hook No. 5	570.31 570.97	+0.09 +0.10	−0.95 −1.27	−2.98 −3.78	−5.44 −5.83	−12.70 −13.20	50 460	−7.1	303.9	31.8	−0.009	Scale—fairly smooth, greenish gray over brown, adherent.
Key West No. 7	560.31 557.54	+0.08 +0.08	−0.01 −0.02	−0.14 −0.14	−0.32 −0.33	−3.64 −3.74	51 700	−4.8	328.8	31.7	−0.006	Film—smooth, purplish brown, green at edges, adherent.
La Jolla No. 9	561.62 565.95	+0.50 +0.46	+0.35 +0.30	−0.04 −0.03	−0.60 −0.54	−3.82 −3.80	51 670	−4.9	653.8	32.5	−0.031	Film—smooth, green (scale) over brown, adherent.
State College No. 3	572.69 573.60	−0.06 −0.07	−0.26 −0.27	−0.61 −0.64	−1.04 −1.17	−4.37 −4.35	51 880	−4.5	625.1	31.4	+0.003	Film—smooth, black, adherent.
Phoenix No. 8	560.51 562.50	+0.09 +0.11	+0.08 +0.08	+0.09 +0.11	+0.07 +0.08	−1.10 −1.15	52 520	−3.3	425.4	31.9	−0.022	Film—smooth, yellowish gray, adherent.
Storage, No. 10	......	......	......	......	......	......	52 710	−3.0	872.8	31.9	−0.022	
Material Q—Copper-Nickel-Tin Alloy (70 Cu, 29 Ni, 1 Sn)												
Pittsburgh No. 1	543.95 544.16	0.00 −0.05	−2.77 −2.35	−8.23 −5.68	....	....	......	......	......	......	......	
Altoona No. 2	549.83 553.56	−0.74 −0.69	−2.66 −2.65	−6.85 −7.01	−17.99 −18.16	−26.20 −26.30	69 900	−9.0	175.7	23.8	+0.016	Film—slightly rough, black with large gray-brown areas, adherent.
New York City No. 4	541.46 546.45	−1.34 −1.35	−4.40 −4.41	−10.27 −10.26	−17.55 −17.66	−25.16 −25.16	71 900	−6.4	349.7	23	+0.048	Film—smooth, dark gray and green mottled, adherent.
Rochester No. 6	542.42 542.75	+0.13 +0.17	+0.78 +0.77	+1.22 +1.20	....	....	73 000	−5.0	389.9	24.8	−0.024	
Sandy Hook No. 5	549.01 550.11	+0.10 +0.10	−0.80 −0.79	−2.91 −2.89	−5.51 −5.42	−13.81 −13.74	73 000	−5.0	440.3	25	−0.033	Film—smooth, grayish green, overreddish brown, adherent.
Key West No. 7	545.10 545.07	+0.05 +0.07	−0.01 −0.01	−0.10 −0.09	−0.23 −0.21	−4.44 −4.42	75 900	−1.1	550.3	24.3	−0.004	Film—smooth, reddish brown, green at edges, adherent.
La Jolla No. 9	544.72 544.59	+0.31 +0.33	+0.17 +0.14	−0.23 −0.27	−0.90 −0.88	−3.36 −3.39	76 500	−0.4	524.6	23.8	−0.016	Film—smooth, brown with bright green outer scale, adherent.
State College No. 3	543.55 544.53	−0.11 −0.11	−0.31 −0.31	−0.64 −0.63	−1.14 −1.14	−4.33 −4.39	77 600	+1.0	676.7	22.3	+0.078	Film—smooth, brownish black, adherent.
Phoenix No. 8	544.42 545.23	+0.08 +0.08	+0.05 +0.05	−0.07 −0.06	+0.06 +0.05	−0.96 −0.94	80 900	+5.0	549.3	20.3	+0.16	Tarnish—heavy brownish gray.
Storage, No. 10	......	......	......	......	......	......	78 800	+2.6	572.4	22.3	+0.078	

CHANGES DUE TO ATMOSPHERIC CORROSION

Location	Original Weight, g., 9 by 12-in. Plate Specimens	Actual Change in Weight, g., 9 by 12-in. Plate Specimens					Tensile Properties After 10-yr. Exposure—Specimens as Exposed on Test Racks				Change in Elongation Before Chemical Cleaning, per cent	Description of Corrosion Product Films on 9 by 12-in. Plate Specimens as Removed from Racks
		After 1 yr.	After 3 yr.	After 6 yr.	After 10 yr. Uncleaned	After 10 yr. Chemically Cleaned	Tensile Strength, psi., Average	Change in Tensile Strength, per cent	Standard Deviation, psi., Average	Elongation, per cent, Average		
Material S—Nickel-Copper Alloy (70 Ni, 30 Cu)												
Pittsburgh No. 1	557.73 551.88	−1.14 −1.12	−3.00 −2.94	−6.68 −6.56	......	......	......	......	......	......	......	
Altoona No. 2	555.91 546.07	−1.10 −1.09	−3.88 −3.88	−8.32 −8.31	−16.11 −16.09	−17.30 −17.31	76 700	−3.4	113.0	34.4	+0.033	Film—fairly smooth, black, adherent, sooty.
New York City No. 4	542.23 548.76	−1.61 −1.65	−4.42 −4.45	−8.95 −9.08	−15.98 −16.20	−16.51 −16.71	76 060	−4.2	118.1	36.1	−0.014	Film—smooth, yellowish brown, adherent, with loose powdery surface deposit.
Rochester No. 6	541.65 538.20	−0.16 −0.17	−0.59 −0.60	−1.55 −1.56	......	......	78 350	−1.3	860.5	35.9	−0.008	
Sandy Hook No. 5	541.51 548.25	−0.44 −0.41	−1.87 −1.73	−4.22 −4.14	−7.44 −7.71	−7.75 −8.04	77 160	−2.8	677.1	35.4	+0.005	Film—smooth, brownish gray, adherent.
Key West No. 7	556.84 563.58	−0.10 −0.08	−0.24 −0.23	−0.63 −0.59	−1.18 −1.12	−1.96 −1.89	79 830	+0.6	117.5	35.3	+0.008	Tarnish—smooth, gray green.
La Jolla No. 9	554.44 553.40	+0.15 +0.19	−0.25 −0.23	−0.77 −0.71	−1.53 −1.45	−2.25 −2.19	79 510	+0.2	177.3	35.1	+0.014	Film—smooth, gray green with spots of loose green scale, adherent.
State College No. 3	550.61 543.71	−0.15 −0.16	−0.37 −0.39	−0.76 −0.79	−1.39 −1.40	−1.47 −1.48	79 190	−0.3	617.2	36.0	−0.011	Tarnish—smooth, gray.
Phoenix No. 8	555.35 546.33	+0.02 +0.02	−0.05 −0.05	−0.07 −0.07	−0.24 −0.22	−0.49 −0.47	79 180	−0.3	109.1	35.5	+0.002	Tarnish—smooth, gray.
Storage, No. 10	......	......	......	......	......	......	79 290	−0.1	533.8	35.1	+0.014	
Material T—Commercial Nickel (99+ Ni)												
Pittsburgh No. 1	560.76 564.43	−3.90 −3.86	−10.82 −10.71	−23.05 −22.79	......	......	......	......	......	......	......	
Altoona No. 2	565.49 565.60	−3.83 −3.95	−11.76 −12.04	−24.86 −25.48	−46.44 −47.61	−47.29 −48.50	64 170	−9.7	241.3	35.1	+0.019	Film—slightly rough, mottled dark gray green and black, adherent.
New York City No. 4	561.82 565.53	−5.01 −5.07	−12.48 −12.58	−24.46 −24.45	−40.10 −40.26	−40.48 −40.64	65 140	−8.4	218.4	35.4	+0.011	Film—smooth, mottled gray and green, adherent.
Rochester No. 6	561.22 566.87	−0.53 −0.54	−2.03 −2.06	−5.52 −5.76	......	......	68 600	−3.5	279.4	36.3	−0.013	
Sandy Hook No. 5	561.51 562.28	−0.74 −0.74	−2.36 −2.39	−5.10 −5.14	−9.24 −9.22	−9.37 −9.33	71 370	+0.4	601.7	36.4	−0.016	Film—smooth, dark gray, adherent.
Key West No. 7	564.13 559.16	−0.14 −0.14	−0.41 −0.41	−0.75 −0.75	−1.34 −1.35	−1.49 −1.49	72 480	+1.9	875.0	36.7	−0.025	Tarnish—smooth, gray adherent.
La Jolla No. 9	563.82 563.36	+0.08 +0.10	−0.19 −0.19	−0.53 −0.57	−0.99 −1.01	−1.39 −1.37	72 530	+2.0	802.6	36.4	−0.016	Tarnish—smooth, gray spotted with many gray-green scale spots.
State College No. 3	566.06 562.88	−0.12 −0.12	−0.39 −0.38	−0.97 −0.98	−1.90 −1.91	−1.94 −1.96	73 040	+2.7	651.5	36.6	−0.022	Tarnish—smooth and bright, gray.
Phoenix No. 8	564.94 561.31	00.00 0.00	−0.06 −0.06	−0.09 −0.10	−0.28 −0.30	−0.36 −0.39	72 240	+1.6	725.5	36.3	−0.013	Tarnish—bright, smooth, light gray.
Storage, No. 10	......	......	......	......	......	......	72 950	+2.5	794.0	36.7	−0.025	

CHANGES DUE TO ATMOSPHERIC CORROSION

Location	Original Weight, g., 9 by 12-in. Plate Specimens	Actual Change in Weight, g., 9 by 12-in. Plate Specimens					Tensile Properties After 10-yr. Exposure—Specimens as Exposed on Test Racks				Change in Elongation Before Chemical Cleaning, per cent	Description of Corrosion Product Films on 9 by **12-in.** Plate Specimens as Removed from Racks
		After 1 yr.	After 3 yr.	After 6 yr.	After 10 yr. Uncleaned	After 10 yr. Chemically Cleaned	Tensile Strength, psi., Average	Change in Tensile Strength, per cent	Standard Deviation, psi., Average	Elongation, per cent, Average		
					Material U—**Chemical Lead** (99.92 **Pb**, 0.06 **Cu**)							
Pittsburgh No. 1	714.93 717.95	+0.14 +0.03	−0.21 −0.24	−1.04 −1.37	....	....	....	....	....	....	......	
Altoona No. 2	713.98 711.36	−0.21 −0.24	−0.66 −0.76	−1.42 −1.67	−2.80 −3.31	−9.52 −12.12	2 370	−8.2	30.55	40.8	+0.064	Film—black, slightly loose, sooty.
New York City No. 4	715.64 713.77	−0.04 −0.11	−0.47 −0.89	−1.05 −1.52	−1.83 −2.27	−6.65 −6.90	2 365	−8.4	30.96	38.6	+0.114	Film—Smooth, dark gray, adherent.
Rochester No. 6	737.46 738.12	+0.04 0.00	0.00 −0.03	−0.04 −0.12	....	....	2 370	−8.2	26.46	34.8	+0.20	
Sandy Hook No. 5	704.78 701.72	−0.13 −0.17	−1.19 −1.11	−2.34 −2.32	....	....	2 308	−10.6	71.15	37.1	+0.148	
Key West No. 7	720.15 723.72	−0.50 −0.45	−1.19 −1.24	−2.89 −2.97	−5.12 −5.31	−8.55 −9.00	2 353	−8.8	28.95	41.8	+0.041	Film—smooth, very light gray, adherent.
La Jolla No. 9	717.24 716.29	+0.29 +0.33	+0.10 −0.14	−0.64 −0.60	−1.79 −1.65	−6.45 −6.68	2 392	−7.3	23.76	35.2	+0.19	Film—smooth, mottled gray and white, adherent.
State College No. 3	718.49 718.33	+0.02 −0.10	−0.50 −0.64	−0.93 −1.18	−1.77 −2.37	−7.68 −7.60	2 383	−7.7	68.10	38.8	+0.11	Film—smooth light gray with white spots, adherent.
Phoenix No. 8	712.98 711.92	−0.04 −0.03	−0.17 −0.20	−0.37 −0.36	−0.62 −0.67	−4.24 −3.45	2 410	−6.6	37.86	39.8	+0.08	Tarnish—smooth, gray.
Storage, No. 10	......	......	......	......	......	......	2 353	−8.6	46.90	42.5	+0.025	
					Material Z—**Aluminum-Magnesium-Silicon** (98 **Al**, 0.5 **Mg**, 1 **Si**)							
Pittsburgh No. 1	160.61 160.19	+0.22 +0.21	+0.27 +0.28	+0.38 +0.54	....	....	....	....	....	....	......	
Altoona No. 2	162.66 163.26	+0.25 +0.22	+0.56 +0.47	+0.86 +0.74	+1.44 +1.25	−2.32 −2.37	36 200	−9.5	317.6	12.0	+0.49	Film—fairly smooth, black, adherent, sooty.
New York City No. 4	163.05 167.63	+0.32 +0.33	+0.41 +0.39	+0.64 +0.59	+0.64 +0.55	−2.54 −2.87	37 520	−6.2	554.5	13.9	+0.41	Film—slightly rough, dark gray, adherent, sooty.
Rochester No. 6	160.89 159.59	+0.11 +0.09	+0.21 +0.20	+0.37 +0.38	....	....	39 750	−0.6	302.3	14.1	+0.41	
Sandy Hook No. 5	169.40 168.85	+0.22 +0.21	+0.33 +0.27	+0.34 +0.27	+0.40 +0.35	−0.36 −0.36	38 820	−2.9	613.6	14.5	+0.39	Film—slightly rough, mottled, light gray.
Key West No. 7	166.85 167.80	+0.13 +0.09	+0.16 +0.14	+0.30 +0.25	+0.35 +0.30	−0.18 −0.17	41 150	+2.9	317.4	12.8	+0.46	Tarnish—gray with many small white specks slightly rough.
La Jolla No. 9	162.34 162.32	+0.78 +0.85	+0.96 +1.00	+1.75 +1.68	+2.94 +2.74	−2.46 −2.37	31 130	−22.1	159.1	4.8	+0.79	Film—rough, white with spot of scale, nonadherent.
State College No. 3	160.31 167.56	−0.01 −0.01	−0.03 −0.02	−0.05 −0.04	−0.07 −0.07	−0.16 −0.16	41 925	+4.8	863.7	20.8	+0.13	Tarnish—smooth, gray.
Phoenix No. 8	161.14 159.64	+0.02 +0.03	0.00 +0.01	+0.02 +0.02	+0.08 +0.07	−0.02 −0.02	43 450	+8.7	557.7	17.0	+0.28	Tarnish—smooth, gray.
Storage, No. 10	......	......	......	......	......	......	42 715	+6.9	273.0	23.1	+0.03	

CHANGES DUE TO ATMOSPHERIC CORROSION

Location	Original Weight, g., 9 by 12-in. Plate Specimens	Actual Change in Weight, g., 9 by 12-in. Plate Specimens					Tensile Properties After 10-yr. Exposure—Specimens as Exposed on Test Racks				Change in Elongation Before Chemical Cleaning, per cent	Description of Corrosion Product Films on 9 by 12-in. Plate Specimens as Removed from Racks
		After 1 yr.	After 3 yr.	After 6 yr.	After 10 yr. Uncleaned	After 10 yr. Chemically Cleaned	Tensile Strength, psi., Average	Change in Tensile Strength, per cent	Standard Deviation, psi., Average	Elongation, per cent, Average		
Material AA—Commercial Zinc (Prime Western Grade)												
Pittsburgh No. 1	445.30 469.72	−2.21 −2.30	−6.41 −6.62	−14.32 −15.02	....	....	....	....	....	....	......	
Altoona No. 2	443.15 442.92	−2.67 −2.73	−8.49 −8.63	−18.66 −19.08	−38.23 −39.13	−48.39 −48.87	22 600	−24.4	340.2	18.9	+0.44	Film—rough, dark gray, adherent.
New York City No. 4	440.55 447.63	−3.27 −3.23	−9.61 −9.48	−21.35 −21.10	−41.05 −40.97	−48.95 −49.55	23 100	−22.9	220.8	28.9	+0.15	Film—rough, grayish white, adherent.
Rochester No. 6	467.35 466.77	−0.20 −0.24	−0.97 −1.12	−2.85 −3.29	....	....	26 800	−10.6	179.0	27.6	+0.19	
Sandy Hook No. 5	467.23 466.70	−0.45 −0.45	−2.31 −2.38	−5.27 −5.35	−10.20 −10.17	−14.08 −14.12	27 100	−9.4	180.0	32.3	+0.058	Film—slightly rough, medium gray.
Key West No. 7	438.87 468.10	+0.20 +0.31	+0.04 +0.06	−0.03 0.00	−0.06 −0.03	−5.16 −5.32	28 500	−4.7	149.7	34.3	0	Film—slightly rough, dark gray.
La Jolla No. 9	441.13 468.47	+2.68 +3.10	+4.88 +5.21	+7.29 +7.96	+10.13 +11.20	−16.62 −18.29	25 900	−13.5	365.1	10.2	+0.70	Film—rough, white, speckled, adherent.
State College No. 3	467.25 468.17	−0.06 −0.11	−1.05 −1.07	−2.83 −2.88	−4.22 −5.31	−9.76 −10.64	27 300	−8.7	256.4	32.3	+0.058	Film—smooth, dark gray, adherent.
Phoenix No. 8	468.65 469.38	+0.11 +0.12	+0.04 +0.04	+0.01 +0.04	−0.33 +0.01	−2.40 −2.67	28 500	−4.7	250.0	35.2	−0.027	Film—smooth, dark gray.
Storage, No. 10	......	......	......	......	......	......	29 100	−2.9	211.9	36.7	−0.069	
Material BB—Commercial Zinc (High Grade)												
Pittsburgh No. 1	447.17 448.80	−2.35 −2.34	−6.93 −7.02	−16.30	....	....	....	....	....	....	......	
Altoona No. 2	445.89 446.97	−2.84 −2.80	−8.86 −8.90	−20.97 −20.94	−39.77 −41.38	−49.32 −49.30	13 900	−24.1	268.6	43.6	+0.25	Film—rough, dark gray, adherent (speckled with soot).
New York City No. 4	447.03 445.93	−3.65 −3.60	−10.49 −10.40	−22.22 −23.10	−43.01 −42.65	−51.70 −51.52	14 200	−22.2	156.9	52.4	+0.104	Film—rough, light gray, adherent.
Rochester No. 6	445.98 446.57	−0.26 −0.26	−1.31 −1.31	−4.00 −4.01	....	....	16 600	−4.0	50.00	53.8	+0.08	
Sandy Hook No. 5	446.31 447.16	−0.49 −0.59	−2.58 −2.84	−5.59 −6.00	−10.79 −11.37	−13.98 −14.52	16 800	−8.4	170.8	52.8	+0.09	Film—smooth, medium gray, adherent.
Key West No. 7	450.16 449.96	+0.13 +0.13	−0.19 −0.19	−0.53 −0.48	−1.09 −0.94	−4.32 −4.83	17 500	−4.4	93.66	56.3	+0.03	Film—rough, medium gray.
La Jolla No. 9	448.72 448.76	+2.98 +3.10	+4.89 +4.69	+6.85 +6.59	+8.89 +9.93	−15.41 −16.59	16 100	−12.0	90.35	30.5	+0.46	Scale—rough, white, somewhat speckled, adherent.
State College No. 3	448.13 449.37	−0.27 −0.27	−1.30 −1.28	−3.26 −3.17	−5.99 −5.83	−8.79 −8.68	17 100	−6.3	94.88	53.4	+0.07	Film—smooth, medium gray, adherent.
Phoenix No. 8	445.33 448.39	+0.08 +0.08	0.00 0.00	+0.17 +0.09	+0.05 −0.08	−1.60 −1.98	17 700	−3.1	35.65	57.3	+0.02	Tarnish—mottled dark gray.
Storage, No. 10	......	......	......	......	......	......	18 000	−1.7	73.73	57.0	+0.02	

CHANGES DUE TO ATMOSPHERIC CORROSION

Location	Original Weight, g., 9 by 12-in. Plate Specimens	Actual Change in Weight, g., 9 by 12-in. Plate Specimens					Tensile Properties After 10-yr. Exposure—Specimens as Exposed on Test Racks				Change in Elongation Before Chemical Cleaning, per cent	Description of Corrosion Product Films on 9 by 12-in. Plate Specimens as Removed from Racks
		After 1 yr.	After 3 yr.	After 6 yr.	After 10 yr. Uncleaned	After 10 yr. Chemically Cleaned	Tensile Strength, psi., Average	Change in Tensile Strength, per cent	Standard Deviation, psi., Average	Elongation, per cent, Average		
Material CC—Lead-Antimony Alloy (1 per cent Sb)												
Pittsburgh No. 1	743.08 704.98	0.00 +0.04	−0.21 −0.14	−0.71 −0.93	....	....	....	....	....	....	......	
Altoona No. 2	713.45 711.74	−0.15 −0.16	−0.56 −0.41	−1.46 −1.10	−2.23	−8.65	3 075	−11.7	12.58	42.7	+0.057	Film—smooth, black, adherent, sooty.
New York City No. 4	712.83 709.90	−0.02 0.00	−0.26 −0.24	−0.68 −0.78	−1.13 −1.26	−3.99 −6.04	3 175	−9.1	18.03	43.2	+0.046	Film—very dark gray.
Rochester No. 6	708.67 706.53	+0.03 +0.02	+0.08 +0.06	+0.08 +0.11	....	....	3 130	−10.1	19.15	42.3	+0.066	
Sandy Hook No. 5	714.46 712.77	−0.04 −0.16	−0.56 −0.54	−1.28 −1.29	....	....	3 130	−10.1	31.09	43.6	+0.037	
Key West No. 7	713.08 713.61	−0.33 −0.34	−0.66 −0.89	−1.73 −1.98	−3.44 −3.75	−6.52 −10.04	3 055	−12.3	41.13	40.9	+0.097	Film—smooth, very light gray.
La Jolla No. 9	710.53 712.43	+1.37 +0.77	+1.17 +0.74	+1.09 +1.23	−0.09 +0.13	−10.05 −9.08	3 050	−12.4	75.28	39.0	+0.139	Film—fairly smooth, white, somewhat mottled.
State College No. 3	714.06 715.01	−0.21 −0.20	−0.53 −0.59	−1.07	−1.65	−5.23	3 145	−9.7	29.30	42.6	+0.057	Film—very smooth, dark gray.
Phoenix No. 8	711.18 711.21	−0.05 −0.03	−0.18 −0.17	+0.30 −0.07	−0.56 −0.57	−4.70 −4.09	3 035	−12.8	41.53	40.6	+0.103	Film—very smooth, medium gray, still shows traces of initial tarnish.
Storage, No. 10	......	......	......	......	......	......	3 248	−6.7	13.80	48.4	−0.068	
Material EE—Commercial Tin (99.85 + Sn)												
Pittsburgh No. 1	464.38 463.64	+0.33 +0.32	+0.39 +0.66	...... +1.45	....	....	....	....	....	....	......	
Altoona No. 2	469.01 462.55	+0.45 +0.46	+1.27 +1.27	+2.42 +2.57	+3.84 +4.27	−15.83 −15.47	2 803	−25.4	95.61	47.4	+0.213	Film—smooth, black with white areas, adherent, sooty.
New York City No. 4	470.83 470.43	+0.50 +0.52	+0.89 +0.92	+1.48 +1.49	+2.84 +2.81	−12.39 −12.11	2 927	−22.1	63.79	56.3	+0.066	Film—smooth, very dark, speckled with white, adherent.
Rochester No. 6	464.92 466.23	+0.33 +0.32	+0.53 +0.55	+0.87 +0.88	....	....	2 897	−22.9	62.21	55.3	+0.082	
Sandy Hook No. 5	470.64 472.62	+1.04 +1.00	+1.24 +1.19	+2.20 +2.07	....	....	2 708	−28.0	47.94	34.0	+0.436	
Key West No. 7	465.64 467.58	+0.43 +0.47	+1.12 +1.17	+2.74 +2.82	+5.26 +5.49	−21.59 −22.22	2 775	−26.2	97.77	55.7	+0.076	Film—smooth, white speckled, adherent
La Jolla No. 9	470.68 468.04	+1.50 +1.45	+2.40 +2.16	+3.87 +3.71	+6.09 +5.80	−24.00 −23.03	2 413	−35.8	183.0	18.0	+0.702	Scale—rough, white and somewhat mottled.
State College No. 3	469.23 471.13	+0.19 +0.17	+0.22 +0.21	+0.25 +0.23	+0.35 +0.42	−4.54 −4.57	3 038	−19.2	80.83	66.3	−0.099	Film—smooth, very light gray.
Phoenix No. 8	467.84 470.34	+0.14 +0.14	+0.19 +0.20	+0.34 +0.93	+0.59 +1.07	−2.93 −3.71	2 918	−22.4	43.97	69.6	−0.154	Tarnish—smooth, white mottled.
Storage, No. 10	......	......	......	......	......	......	3 332	−11.4	77.19	66.5	−0.102	

CHANGES DUE TO ATMOSPHERIC CORROSION

Location	Original Weight, g., 9 by 12-in. Plate Specimens	Actual Change in Weight, g., 9 by 12-in. Plate Specimens					Tensile Properties After 10-yr. Exposure—Specimens as Exposed on Test Racks				Change in Elongation Before Chemical Cleaning, per cent	Description of Corrosion Product Films on 9 by 12-in. Plate Specimens as Removed from Racks
		After 1 yr.	After 3 yr.	After 6 yr.	After 10 yr. Uncleaned	After 10 yr. Chemically Cleaned	Tensile Strength, psi., Average	Change in Tensile Strength, per cent	Standard Deviation, psi., Average	Elongation, per cent, Average		
Material FF—Aluminum-Coated Duralumin												
Pittsburgh No. 1	167.76 171.26	+0.20 +0.18	+0.18 +0.14	+0.12 +0.11	....	....	....	....	....	....	......	
Altoona No. 2	167.84 170.64	+0.27 +0.24	+0.37 +0.39	+0.50 +0.47	+0.80 +0.82	−2.67 −2.53	58 790	0.0	422.7	16.3	+0.163	Film—smooth, black, sooty.
New York City No. 4	169.40 173.25	+0.35 +0.32	+0.26 +0.24	+0.17 +0.16	−0.24 −0.23	−2.52 −2.19	58 835	+0.1	180.6	17.8	+0.087	Film—fairly smooth, dark gray, adherent, sooty.
Rochester No. 6	169.77 169.48	+0.12 +0.11	+0.29 +0.22	+0.66 +0.38	....	....	56 990	−3.1	695.1	14.6	+0.251	
Sandy Hook No. 5	171.53 173.09	+0.20 +0.23	+0.25 +0.26	+0.14 +0.16	+0.04 +0.06	−0.63 −0.51	57 180	−2.7	186.4	16.3	+0.164	Film—fairly smooth, light gray, speckled, adherent.
Key West No. 7	172.96 167.17	+0.09 +0.07	+0.14 +0.12	+0.20 +0.18	+0.21 +0.19	−0.13 −0.14	60 295	+2.5	339.8	18.7	+0.041	Tarnish—slightly rough, gray speckled with white film.
La Jolla No. 9	172.22 170.88	+0.62 +0.63	+0.55 +0.60	+1.13 +1.14	+2.22 +2.24	−1.67 −1.56	58 730	−0.1	100.1	16.4	+0.158	Tarnish—speckled, gray with loose white scale spots.
State College No. 3	169.45 171.61	−0.01 +0.09	−0.03 +0.07	−0.05 +0.05	−0.09 +0.01	−0.15 −0.05	59 590	+1.3	637.5	19.0	+0.025	Tarnish—smooth, light gray.
Phoenix No. 8	170.06 168.98	+0.03 +0.03	+0.01 +0.01	+0.03 +0.03	+0.05 +0.05	−0.01 −0.01	59 300	+0.9	993.8	18.0	+0.051	Tarnish—smooth, light gray.
Storage, No. 10	......	......	......	......	......	......	59 935	−1.9	944.4	19.5	0	
Material HH—Commercial Zinc (Special High Grade 99.9% Zn)												
Pittsburgh No. 1	446.02 430.83	−2.52 −3.29	−7.59 −7.98	−17.49 −17.30	....	....	....	....	....	....	......	
Altoona No. 2	436.54 435.41	−2.92 −2.83	−9.15 −9.03	−20.72 −20.85	−40.12 −39.89	−44.66 −44.45	12 881	−29.5	127.2	45.5	+0.118	Film—fairly rough, medium gray, overcast with soot.
New York City No. 4	432.61 432.64	−3.68 −3.66	−10.81 −10.77	−23.58 −23.53	−42.91 −42.78	−48.82 −48.74	14 674	−19.7	563.0	49.8	+0.034	Film—somewhat rough, light gray.
Rochester No. 6	435.38 433.95	−0.22 −0.22	−1.25 −1.24	−4.12 −4.15	....	....	16 517	−9.6	165.8	57.4	−0.112	
Sandy Hook No. 5	434.37 433.67	−0.44 −0.46	−2.57 −2.60	−5.83 −5.89	−11.04 −11.14	−13.80 −13.90	16 741	−8.4	110.6	54.9	−0.063	Film—fairly smooth, light gray.
Key West No. 7	435.58 435.64	+0.14 +0.22	−0.18 −0.08	−0.54 −0.42	−1.08 −0.97	−3.90 −3.79	17 520	−4.2	160.3	60.8	−0.178	Film—slightly rough, light gray with small white specks.
La Jolla No. 9	435.85 434.91	+2.53 +2.40	+3.96 +3.75	+5.62 +5.27	+7.10 +7.07	−12.55 −11.95	16 406	−10.2	151.7	30.4	+0.410	Film—rough, gray speckled with white.
State College No. 3	432.89 430.75	−0.13 −0.07	−1.32 −1.28	−3.60 −3.45	−6.64 −6.51	−9.15 −9.08	17 237	−5.7	245.0	63.2	−0.224	Film—smooth, light gray.
Phoenix No. 8	442.17 439.06	+0.09 +0.09	0.00 −0.01	−0.05 −0.05	−0.22 −0.06	−1.18 −1.17	17 956	−1.8	112.3	61.9	−0.19	Film—smooth, medium gray.
Storage, No. 10	......	......	......	......	......	......	16 686	−8.7	245.8	64.1	−0.242	

REPORT OF COMMITTEE B-4

ON

ELECTRICAL-HEATING, ELECTRICAL-RESISTANCE AND ELECTRIC-FURNACE ALLOYS

Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys held three meetings during the year: in Atlantic City, N. J., on June 23 and 24, 1942, and in New York, N. Y., on October 29 and 30, 1942, and on February 18 and 19, 1943.

The committee consists of 54 members, of whom 28 are classified as producers, 20 as consumers, and 6 as general interest members.

A special committee has given consideration to the subject of substitute alloys for the present electrical-resistance alloys which contain critical materials. The conclusions reached have indicated that there are no new substitute alloys which have the electrical properties and corrosion resistance required for most applications.

I. NEW TENTATIVE STANDARDS

The committee recommends that the following methods of test be accepted for publication as tentative:[1]

Tentative Methods of Test for:

Density of Fine Wire and Ribbon for Electronic Devices,
Effect of Controlled Atmospheres Upon Alloys in Electric Furnaces, and
Life Test of Electrical Contact Materials.

II. EDITORIAL CHANGE IN TENTATIVE STANDARD

The committee recommends the following editorial change in the Tentative Method of Test for Temper of Strip and Sheet Metals for Electronic Devices (Spring-Back Method) (B 155 – 41 T):[2]

Section 2 (c).—Change to read as follows by the addition of the italicized words:

> (*c*) Scale.—A quadrant scale with its center in the axis of rotation and capable of rotation about this axis through an angle not less than 35 deg. as shown in Fig. 1. *A suitable clamp shall be provided to hold the scale firmly while the test is being made.* The scale shall be capable of being clamped rigidly to the frame, shall be divided into intervals of not more than 1 deg., and shall read 90 deg. at the initial position and zero at the position of maximum deflection of the specimen. A stop shall be provided on the scale so that the clamp with the specimen cannot be rotated more than 90 deg.

Section 4 (e).—Change to read as follows by the addition of the italicized words:

> (*e*) Observation of Spring-Back.—After completing the bending operation, the clamp shall be returned *immediately* until contact of the specimen with the bending edge is just lost *and then re-established.* The angle between the extreme position and that where contact with the bending edge is just made, shall be recorded as the spring-back.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 54 members; 32 members returned their ballots, with the results shown in Table I.

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[1] These methods were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part I, pp. 312, 308, and 315, respectively.

[2] 1942 Book of A.S.T.M. Standards, Part I, p. 1440.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Test for Density of Fine Wire and Ribbon for Electronic Devices	28	4	0
Test for Effect of Controlled Atmospheres Upon Alloys in Electric Furnaces	27	5	0
Life Test of Electrical Contact Materials	28	4	0
II. EDITORIAL CHANGE IN TENTATIVE STANDARD			
Test for Temper of Strip and Sheet Metals for Electronic Devices (Spring-Back Method) (B 155 - 41 T)	30	2	0

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Life Tests (J. W. Harsch, chairman).—The interlaboratory life tests on heater wire, which have been made to check equipment and test procedure, have shown good agreement in results by two laboratories. The other two laboratories have not been able to make the tests because of pressure of war work, and further study has been postponed for the present.

The subcommittee has under consideration revisions in the Standard Specifications for Drawn or Rolled Alloy, 80 per cent Nickel, 20 per cent Chromium for Electrical Heating Elements (B 82 - 41) and for Drawn or Rolled Alloy, 60 per cent Nickel, 15 per cent Chromium, and Balance Iron (B 83 - 41).

Subcommittee V on Wrought and Cast Alloys for High Temperature Use (J. W. Harsch, chairman).—This subcommittee has continued its study of the significant properties and methods of test of alloys for use at temperatures over 1000 F. The Proposed Specifications for Chromium-Nickel-Iron Alloy Castings for High-Temperature Service in Electric Furnaces, which were published as information in Appendix I to the 1942 annual report, have been reviewed and modified and are now being given further study. Work is planned to obtain more complete information on the properties and methods of test specified.

Subcommittee VII on Thermostat Metals (P. H. Brace, chairman).—A Proposed Method of Measurement of Equivalent Yield Stress for Thermostat Metals has been prepared to indicate the yield stress in bending at any desired temperature, and is published as information as an Appendix to this report. This method provides for the calculation of yield stress by the usual beam formula from the moment required to produce a specified amount of plastic bending. Interlaboratory tests will be made to try the method on a more extensive scale than has been practicable.

Subcommittee VIII on Metallic Materials for Radio Tubes and Incandescent Lamps (Stanton Umbreit, chairman).—The Tentative Method of Test for Temper of Strip and Sheet Metals for Electronic Devices (Spring-Back Method) (B 155 - 41 T) has been reviewed and revised in minor details.

A new Method of Test for Density of Fine Wire has been prepared, which gives more consistent results than other methods in use, and is being submitted for publication as tentative.

Work is under way on an investigation of the testing and grading of powders used in radio tube production, and methods of test for tungsten wire for glass seals.

Subcommittee IX on Methods of Test for Alloys in Controlled Atmospheres (P. H. Brace, chairman).—The Proposed Method of Test for the Effect of Controlled Atmospheres Upon Alloys in Electric Furnaces, which was printed as information in the 1942 annual report, has been revised in minor details and is being submitted for publication as tentative.

Further test work is under way to determine the effect of various controlled atmospheres on heat-resisting alloys, with particular reference to evaluating the performance of such materials in the cast state and to compositions of lower alloy content. These tests are of particular interest because of the need for conservation of critical metals.

Subcommittee X on Contact Materials (F. E. Carter, chairman).—The Method for Life Test of Electrical Contact Materials has been given a trial during the past year and has been modified in minor details. The revision makes the method more general so that it may be applied to various materials under suitable operating conditions. Test details are included for fine silver and will be prepared for other contact materials. The method as revised is being submitted for publication as tentative.

A study of the cause of rebound of contacts during test is being made in an effort to eliminate rebound which apparently is one factor responsible for the sticking of contacts during test.

A recommended list of solid contact rivets has been made, to reduce the number of sizes to a minimum, and has been submitted to the National Electrical Manufacturers Assn. for publication as a standard. At the suggestion of N.E.M.A., a few additional sizes were added, and it is hoped that the list of standard sizes will soon be published.

At the request of N.E.M.A. a suggested list of standard sizes of pointed-head rivets is being prepared. Information is being gathered for standardization of sizes of projection welding contacts.

An abstract bibliography of about 800 references to articles relating to the testing of contact materials has been prepared. This is probably the most complete list on this subject that has been compiled and should be very valuable. It is planned to have this list published by the Society.[3]

This report has been submitted to letter ballot of the committee which consists of 54 members; 32 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

DEAN HARVEY,
Chairman.

F. E. BASH,
Secretary.

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[3] The "Bibliography and Abstracts on Electrical Contacts" was published in January, 1944, and is available as a separate publication.

PROPOSED METHOD OF MEASUREMENT OF EQUIVALENT YIELD STRESS OF THERMOSTAT METALS[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method is intended for the evaluation of the equivalent yield stress of thermostat metal of usual commercial sizes in the form of (*1*) flat strips 0.025 in. and over in thickness, or (*2*) helical coils. No restrictions are placed on test temperatures.

NOTE 1.—In this method the equivalent yield stress is determined independently of the flexivity and the elastic modulus, which later changes rapidly with temperature in the case of the nickel-iron alloys and certain others frequently used as components of thermostat metals.

NOTE 2.—Other properties of interest can also be measured by slightly modifying the apparatus and basic procedures described in this method. These modifications may be dealt with at a later date.

Equivalent Yield Stress

2. (*a*) Equivalent yield stress is calculated from the applied bending moment and the dimensions of the specimen by the following formula:

$$S = \frac{M}{WT^2}$$

where:

S = equivalent yield stress as of outside fibers in pounds per square inch,
M = yield moment in pounds per inch,
W = width of specimen in inches, and
T = thickness in inches.

(*b*) Yield moment is the applied bending moment required to produce a specified permanent bend with reference to the initial curvature of the specimen at the temperature of test.

(*c*) *Direction of Applied Bending Moments.*—The high expansion side shall be loaded in tension or in compression as agreed upon, since the direction of loading affects the value of the resulting equivalent yield stress.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Heating Device.*—A fluid bath or other means capable of heating the entire specimen and the immediate supporting members so that throughout the specimen during the course of any one test the temperature can be held within such limits that the deflection due to thermostat action will be less than 25 per cent of the specified permanent bend (Section 2 (*b*)). However, the test temperature shall be kept within plus or minus 5 C. of the nominal test temperature.

(*b*) *Specimen Supports.*—Rotatable clamps or other means for supporting the specimen at both ends and applying to it a measured bending moment which shall be uniform over the gage length of the specimen.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys. Published as information, June, 1943.

This proposed method, while in use, has not been formally adopted. When formally approved, it will be incorporated in the Standard Methods of Testing Thermostat Metals (B 106 – 40), 1942 Book of A.S.T.M. Standards, Part I, p. 877.

(*c*) *Gage Marks.*—Gage marks or suitable fixtures placed on the specimen to define the gage length and so located with reference to the specimen supports that measurements of flexure will not be subject to significant error due to any constraint or other action on the specimen by the fixtures used for supporting it or for applying the bending moment.

(*d*) *Flexure Indicator.*—Suitable mechanical, optical, or other means for measuring (*1*) total flexure between gage points produced by the applied bending moment with an error not exceeding plus or minus 2 per cent of the indicated flexure, and (*2*) permanent change in the relative positions of the gage points resulting from the application of the test moment within the following permissible variations:

> (*1*) *Flat Strips.*—For flat strips the permissible variation of the measurement of the position of a point on the surface of the specimen midway between the gage marks relative to the straight line passing through the gage marks shall not exceed 0.0005 in. per in. of gage length.
>
> (*2*) *Helical Coils.*—For helical coils the permissible variation of the measurement of the relative angular positions of the gage points shall not exceed 0.03 deg. per turn of the helix between gage points.

(*e*) *Loading Mechanism.*—Weighted levers or other means for applying and maintaining a constant measured bending moment which shall be uniform throughout the gage length of the specimen.

(*f*) *Temperature Measuring Apparatus.*—Calibrated thermocouples and potentiometer capable of determining the temperature within plus or minus 5 C. and a sufficient sensitivity and responsiveness to meet the bath stability requirements prescribed in Paragraph (*a*). The thermocouples shall be attached to the specimen or so closely adjacent that the measured temperatures are the same as those of the specimen.

Test Specimens

4. (*a*) *Flat Strips.*—The test specimen shall be in the form of a flat strip whose minimum thickness is not less that 0.025 in. and which displays no significant initial irregularity of curvature. The maximum thickness shall not exceed the minimum thickness by more than 1 per cent of the minimum thickness. Thickness shall be measured to plus or minus 0.0001 in. by means of a screw micrometer or equivalent device. The width shall not be less than 5 times nor more than 10 times the thickness and shall be measured to an accuracy of plus or minus 0.25 per cent. The maximum width shall not exceed the minimum width by more than 0.5 per cent of the minimum width. The length shall be such as to allow a distance between gage points of not less than 10 times nor more than 15 times the mean width. The distance between gage points shall be measured accurately to the nearest 0.25 per cent.

(*b*) *Helical Coils.*—Helical coils shall be used for material less than 0.025 in. in thickness but may be used for thicker material. The test specimen shall be wound on a mandrel to form a circular helix whose pitch and inside diameter over the gage length shall be uniform to within plus or minus 5 per cent and plus or minus 2 per cent, respectively, of the mean values. The helix shall be wound with the high-expansion side of the material either inside or outside as specified. The mean diameter shall be not less than 20 times the thickness. The helix shall be so wound that there will be no contact between adjacent turns for any combination of test moment and temperature. The maximum spacing between turns shall not exceed 10 per cent of the width of the test specimen. The number of turns between gage points shall be 10 or more. When this requirement is met it is usually possible to fasten the specimen to the supports so that the gage length may be regarded as that portion of the specimen between the inside edges of the clamps.

Preparation of Specimens

5. (*a*) After being cut or sheared from the sample, the test specimen shall be finished to size by careful machining or filing. An amount of material extending a distance not less than twice the thickness shall be removed along the edge, to eliminate material damaged by the preliminary shaping.

(*b*) When the specimen has been finished to size, any necessary reference marks shall be made (by such means as a sharp drill, scribing tool, or milling cutter) and their relative locations determined and recorded. Center punches shall not be used because of the distortion produced.

Stabilization of Specimen

6. After all preparatory work on the test specimen has been completed, it shall be subjected to a stabilizing heat treatment to relieve internal stresses. This treatment may consist in heating the specimen, while free to bend, for a prescribed time and temperature. The details of the stabilizing procedure will depend upon the characteristics of the thermostat metal being tested and shall be as agreed upon by the manufacturer and the purchaser.

Procedure

7. (*a*) The specimen shall be mounted on its supports. A small moment shall be applied and then removed at a low rate. If appreciable friction or a variable zero position is apparent with repeated applications of a moment, the cause shall be determined and removed before proceeding with the test.

(*b*) When satisfactory initial conditions have been established, the temperature of the specimen shall then be adjusted to the test value.

(*c*) The relative positions of the gage points shall then be measured and recorded.

(*d*) A small measured test moment shall be applied, recorded, and maintained for a specified time. The moment shall then be removed in such a way as to avoid flexural oscillations of the specimen. The specimen shall be allowed to remain undisturbed in the relaxed condition for a specified time and the gage point positions again measured and recorded. This process shall be repeated with successively greater test moments until the yield moment as defined in Section 2 (*b*) has been reached.

(*e*) A separate test specimen shall be taken for each test temperature. The next higher test temperature shall then be established, the new thermal equilibrium positions of the gage points observed, and recorded, and the new yield moment determined as before.

Calculation and Report

8. (*a*) *Calculation.*—The equivalent yield stress shall be calculated from the yield moment and the dimensions of the specimen according to the formula in Section 2 (*a*).

(*b*) *Report.*—The report shall include the following:

(*1*) Class of thermostat metal,

(*2*) Identification of specimen,

(*3*) Type (flat or helical) and dimensions of test specimen, and location of gage points,

(*4*) Stabilization history of specimen and any other pertinent data as agreed upon by the manufacturer and the purchaser,

(*5*) Equivalent yield stress at each temperature, and

(*6*) Direction of applied bending moment, high expansion side in tension, or high expansion side in compression.

REPORT OF COMMITTEE B-5

ON

COPPER AND COPPER ALLOYS, CAST AND WROUGHT

Committee B-5 on Copper and Copper Alloys, Cast and Wrought, held one meeting during the year: in Atlantic City, N. J., on June 23, 1942.

The following additions and changes in membership have occurred:

R. A. Wilkins, Vice-President, Revere Copper and Brass, Inc., has accepted appointment as chairman of Subcommittees III and IV in place of E. S. Bunn.

Subcommittee XIII—J. E. McGraw appointed chairman, replacing R. G. Thompson, resigned.

New Members:

Werner Finster, representing American Chain and Cable Co., Inc.

E. J. Ednie, representing Duquesne Smelting Corp.

V. T. Malcolm (Chapman Valve Mfg. Co.)

E. F. Hess, representing The Ohio Injector Co.

G. M. Thrasher (R. Lavin and Son, Inc.)

H. B. Knowlton, representing International Harvester Co.

New Representatives:

Allen W. Carpenter, replacing C. H. Mercer (resigned), American Railway Engineering Assn.

L. G. Yoder and Paul C. Beck, replacing L. L. Stott (resigned), Beryllium Corporation of Pennsylvania.

M. J. Donachie, replacing Paul C. Beck (resigned), Beryllium Corporation of Pennsylvania.

W. F. Roeser, replacing W. H. Swanger (deceased), National Bureau of Standards.

H. H. Stout, Jr., U. S. Army Ord. Dept., Frankford Arsenal.

B. J. Sirois, replacing H. H. Stout, Jr., Phelps Dodge Copper Products Corp.

L. B. Barker, additional representative of General Electric Co.

R. W. E. Moore, additional representative of National Electric Products Corp.

W. L. Barth, replacing W. P. Eddy, Jr. (resigned), General Motors Corp.

V. O. Wilkerson, replacing G. A. Cullen (resigned), International Business Machines Corp.

J. J. Malone, replacing J. F. Halpin (resigned), Seymour Mfg. Co.

Lt. Col. L. S. Fletcher has been assigned to duties calling him away from Frankford Arsenal for the time being and his name has been removed from the list of officers representing the Arsenal.

During the year the committee lost by death W. H. Swanger who for years represented the National Bureau of Standards. The committee will feel greatly the loss of his valued contributions to its activities.

At the present time the committee consists of 116 members of whom 100 are voting members; 41 are classified as producers, 42 as consumers, and 17 as general interest members.

RECOMMENDATIONS ACCEPTED BY COMMITTEE E-10 ON STANDARDS

Subsequent to the annual meeting, Committee B-5 presented to the Society through Committee E-10 on Standards the following recommendations:

Revision of Tentative Specifications for:

Phosphor Bronze Rods, Bars, and Shapes (B 139 - 42 T), and

Phosphor Bronze Wire (B 159 - 42 T).

These recommendations were accepted[1] by Committee E-10 on August 24, 1942, and the revised specifications, assigned the A.S.T.M. serial designations indicated above, appear in the 1942 Book of A.S.T.M. Standards, Part I.

On December 11, 1942, Committee E-10 accepted[1] recommendations providing for the inclusion of a reference to the Tentative Specifications for Electrolytic Cathode Copper (B 115 – 41 T) in the following specifications:

Tentative Specifications for:

Cartridge Brass Sheet, Strip, and Disk (B 19 – 42 T),
Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 – 40 T),
Brass Sheet and Strip (B 36 – 41 T),
Leaded Brass Sheet and Strip (B 121 – 41 T),
Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 – 39 T),
Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 42 T),
Cartridge Brass Cartridge Case Cups (B 129 – 40 T),
Gilding Metal Sheet and Strip (B 130 – 41 T),
Gilding Metal Bullet Jacket Cups (B 131 – 40 T),
Miscellaneous Brass Tubes (B 135 – 42 T),
Aluminum-Bronze Sand Castings (B 148 – 42 T),
Aluminum Bronze Sheet and Strip (B 169 – 41 T), and
Copper-Alloy Condenser Tube Plates (B 171 – 42 T).

The revised tentative specifications appear in the 1942 Book of A.S.T.M. Standards, Part I.

On December 14, 1942, Committee E-10 accepted the following Emergency Alternate Provisions which endorse the Emergency Specifications for Fire-Refined Copper for Wrought Products and Alloys (ES – 7) as an alternate grade of copper for use in the specifications indicated:

Designation	Emergency Alternate Provisions In
	Standard Specifications for:
EA – B 12	Copper Bars for Locomotive Staybolts (B 12 – 42)
EA – B 100	Rolled-Copper-Alloy Bearing and Expansion Plates for Bridge and Other Structural Uses (B 100 – 40)
EA – B 103	Phosphor Bronze Sheet and Strip (B 103 – 42)
EA – B 111	Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 42)
	Tentative Specifications for:
EA – B 19	Cartridge Brass Sheet, Strip, and Disks (B 19 – 42 T)
EA – B 22	Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 – 42 T)
EA – B 36	Brass Sheet and Strip (B 36 – 42 T)
EA – B 121	Leaded Brass Sheet and Strip (B 121 – 42 T)
EA – B 122	Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 – 42 T)
EA – B 124	Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 42 T)
EA – B 129	Cartridge Brass Cartridge Case Cups (B 129 – 42 T)
EA – B 130	Gilding Metal Sheet and Strip (B 130 – 42 T)
EA – B 131	Gilding Metal Bullet Jacket Cups (B 131 – 42 T)
EA – B 133	Copper Rods, Bars, and Shapes (B 133 – 42 T)
EA – B 134	Brass Wire (B 134 – 42 T)
EA – B 135	Miscellaneous Brass Tubes (B 135 – 42 T)
EA – B 139	Phosphor Bronze Rods, Bars, and Shapes (B 139 – 42 T)
EA – B 148a	Aluminum-Bronze Sand Castings (B 148 – 42 T)
EA – B 151	Copper-Nickel-Zinc Alloy Rod and Wire (B 151 – 42 T)
EA – B 152	Copper Sheet, Strip, and Plate (B 152 – 41 T)
EA – B 159	Phosphor Bronze Wire (B 159 – 42 T)
EA – B 169	Aluminum Bronze Sheet and Strip (B 169 – 41 T)
EA – B 171	Copper-Alloy Condenser Tube Plates (B 171 – 42 T)

These emergency provisions were published in ASTM BULLETIN, No. 120, January, 1943, and have also been issued in the form of pink stickers for attachment to the standards to which they apply.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee B-5 reported the following results of the letter ballot vote from a committee membership of 96: B 139 and B 159, 72 ballots returned, affirmative 54, negative 0, ballots marked "not voting" 18; B 19, B 22, B 36, B 121, B 122, B 124, B 129, B 130, B 131, B 135, B 148, B 169, and B 171, 59 ballots returned, affirmative 49, negative 0, ballots marked "not voting" 10.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is recommending revisions of eight tentative standards, revisions in three standards for immediate adoption, the adoption as standard of three tentative standards without revision and one with revision. The standards and tentative standards affected are listed in Table I and the revisions recommended are given in detail in the Appendix.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked “Not , Voting’
I. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Brass Sheet and Strip (B 36 - 42 T)	39	4	24
Spec. for Seamless Copper Tubes (B 75 - 41 T)	43	1	23
Spec. for Leaded Brass Sheet and Strip (B 121 - 42 T)	37	4	26
Spec. for Cartridge Brass Cartridge Case Cups (B 129 - 42 T)	37	2	28
Spec. for Gilding Metal Sheet and Strip (B 130 - 42 T)	41	2	24
Spec. for Gilding Metal Bullet Jacket Cups (B 131 - 42 T)	40	1	26
Spec. for Miscellaneous Brass Tubes (B 135 - 42 T)	50	0	22
Spec. for Phosphor Bronze Wire (B 159 - 42 T)	36	2	29
II. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Rolled Copper-Alloy Bearing and Expansion Plates for Bridges and Other Structural Uses (B 100 - 40)	43	1	23
Spec. for Phosphor Bronze Sheet and Strip (B 103 - 42)	41	2	24
Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 42)	45	1	21
III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Copper Pipe, Standard Sizes (B 42 - 41 T) as revised	42	1	24
Spec. for Seamless Copper Tubing, Bright Annealed (B 68 - 42 T) as revised	42	2	23
Spec. for Manganese Bronze Rods, Bars, and Shapes (B 138 - 41 T)	44	1	22
Spec. for Leaded Red Brass (Hardware Bronze) Rods, Bars and Shapes (B 140 - 41 T)	44	1	22

The recommendations in this report have been submitted to letter ballot of the committee which consists of 100 voting members, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Because of war activities, the subcommittees of Committee B-5 have held very few meetings during the year. Progress has been made, however, as reported in the following summary of subcommittee activities.

Subcommittee I on Copper-Zinc Sheet and Strip (G. H. Harnden, chairman) held a meeting in New York, N. Y., on April 22, at which action was taken to recommend revisions in the Tentative Specifications for Brass (B 36 - 42 T), Leaded Brass (B 121 - 42 T), and Gilding Metal (B 130 - 42 T), Sheet and Strip as well as for Gilding Metal Bullet Jacket Cups (B 131 - 42 T), and Cartridge Brass Cartridge Case Cups (B 129 - 42 T), all of which are given in the Appendix.

The Subgroup on Grain Size has completed a preliminary report and expects to finish the investigation before the annual meeting.

The subcommittee has arranged for distribution of tables covering preferred sizes of sheet, strip, and plate. Many feel that decision in this matter should be deferred until after the war, hence no definite acceptance of the idea has been voted by any subcommittee of Committee B-5.

Subcommittee II on Copper-Tin Sheet and Strip (H. V. Schlacks, chairman) is recommending for immediate adoption revisions in the Standard Specifications for Rolled Copper-Alloy Bearing and Expansion Plates for Bridge and Other Structural Uses (B 100 - 40) and for Phosphor Bronze Sheet and Strip (B 103 - 42), as given in the Appendix.

Subcommittee III on Copper-Nickel-Zinc Sheet and Strip (E. S. Bunn, chairman) during the year has reviewed the

Emergency Alternate Federal Specifications for Nickel-Silver (German-Silver) Bars, Plates, Rods, Shapes, Sheets, and Strips (E-QQ-N-321) and the A.S.T.M. Tentative Specifications for Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 - 42 T). It is felt that the latter should be revised and data are being presented to the subcommittee for early action.

Subcommittee IV on Miscellaneous Copper-Base Sheet and Strip Alloys (E. S. Bunn, chairman) has cooperated with the special committee (G. H. Harnden, chairman) giving consideration to revisions in the Tentative Specifications for Beryllium-Copper Alloy Rods, Bars, Sheet, Strip, and Wire (B 120 - 41 T). This special committee held a meeting in New York, N. Y., on April 22, and approved drafts of three specifications for beryllium copper which are intended to replace the present specifications. The new specifications cover (*1*) Sheet and Strip, (*2*) Rods and Bars, and (*3*) Wire. It is planned to have these three specifications submitted for letter ballot at an early date.

An Emergency Alternate Provision (EA - B 152) in the Tentative Specifications for Copper Sheet, Strip, and Plate (B 152 - 41 T) was approved by this subcommittee during the year permitting the use of the Emergency Specifications for Fire-Refined Copper for Wrought Products and Alloys (ES - 7) as an alternate grade of copper for use in the manufacture of copper sheet, strip, and plate, except when the material is to be used for electrical purposes. In view of the committee activities in classification of coppers, the subcommittee decided that these specifications are not ready for adoption as standard.

Subcommittee V on Miscellaneous Copper-Base Wire and Rod Alloys (H. H. Stout, Jr., chairman) completed action through Committee E-10 on Standards on matters considered during the 1942 annual meeting, as mentioned earlier in this report.

The subcommittee is recommending for adoption as standard without revision the Tentative Specifications for Manganese Bronze Rods, Bars, and Shapes (B 138 - 41 T) and for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 - 41 T). The subcommittee further recommends that the Tentative Specifications for Phosphor Bronze Wire (B 159 - 42 T) be revised, as indicated in the Appendix, and continued as tentative.

The proposed Specifications for Copper Bus Bars, Rods, Tubes, and Shapes have been given very careful study so that unanimous agreement of the members of the subcommittee is expected.

Consideration is being given to a change in the chemical requirements for grade B2 of the Tentative Specifications for Phosphor Bronze Rods, Bars, and Shapes (B 139 - 42 T).

The subcommittee has been giving consideration to further revisions in the Tentative Specifications for Phosphor Bronze Wire (B 159 - 42 T) with regard to stress-relief annealing and strain testing of phosphor bronze wire. Changes in physical test requirements in the Tentative Specifications for Aluminum Bronze Rods, Bars, and Shapes (B 150 - 41 T) are also under consideration. The subcommittee has reviewed the Emergency Federal Specifications for Phosphor Bronze and Aluminum Bronze.

Subcommittee VI on Condenser Tubes (G. C. Holder, chairman) is recommending the incorporation of a reference to the Tentative Specifications for Electrolytic Cathode Copper (B 115 - 41 T) in the Standard Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 42) as an alternate material for use in these specifications.

The subcommittee has made an editorial change in the flatness tolerances in the Tentative Specifications for Copper-Alloy Condenser Tube Plates (B 171 – 42 T).

Subcommittee VII on Copper or Deoxidized Copper Tubes (C. A. Hill, chairman) is recommending for adoption as standard the Tentative Specifications for Copper Pipe, Standard Sizes (B 42 – 41 T) with a minor revision as given in the Appendix.

The subcommittee is also recommending the adoption as standard of the Tentative Specifications for Seamless Copper Tubing, Bright Annealed (B 68 – 42 T), revised as indicated in the Appendix, as well as revisions in the Tentative Specifications for Seamless Copper Tubes (B 75 – 41 T) which are to be continued as tentative. Under consideration for these specifications are the diameter limits for hard-drawn tube and the tensile strength requirements. The embrittlement test is also being studied.

Subcommittee VIII on Copper-Alloy Tubes for General Use (Alan Morris, chairman) is recommending a revision in the Tentative Specifications for Miscellaneous Brass Tubes (B 135 – 42 T), as given in the Appendix.

Subcommittee IX on Copper-Base Alloy Forgings (J. J. Kanter, chairman) completed action through Committee E-10 to revise the Tentative Specifications for Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 42 T) as mentioned earlier in this report.

Subcommittee X on Copper-Base Alloys for Sand Castings (G. H. Clamer, chairman), due to the illness of the chairman, did not find it possible to hold a meeting of the subcommittee until May at which time a meeting was held in Washington, D. C. Consequently, several items suggested for consideration last June have not been brought to a conclusion.

This subcommittee has given further consideration to the proposed Specifications for Copper-Silicon and Copper-Silicon-Zinc Alloy Castings, but the final draft has not yet been presented to the main committee.

Based on data and information compiled by members of the special committee and presented in Appendix II of the 1942 report,[2] Subcommittee X prepared proposed emergency alternate provisions in the specifications listed below providing for the use of a cast-to-size test bar known as the Lunkenheimer Control Bar LTB-1:

Tentative Specifications for:

Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 – 42 T),
Tin-Bronze and Leaded Tin-Bronze Sand Castings (B 143 – 42 T),
High-Leaded Tin-Bronze Sand Castings (B 144 – 42 T),
Leaded Red Brass and Leaded Semi Red Brass Sand Castings (B 145 – 42 T),
Leaded Yellow Brass Sand Castings for General Purposes (B 146 – 42 T)

Standard Specifications for:

Castings of the Alloy: Copper 88 per cent, Tin 8 per cent, Zinc 4 per cent (B 60 – 41),
Steam or Valve Bronze Castings (B 61 – 42), and
Composition Brass or Ounce Metal Castings (B 62 – 41).

This recommendation was further considered by the subcommittee at the May meeting and will shortly be submitted to Committee E-10 on Standards.[3]

The subcommittee has under consideration revisions in the Tentative Specifications for Leaded High-Strength Yellow Brass (Manganese Bronze) Sand Castings (B 132 – 41 T).

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[2] "Studies of Machined *versus* As-Cast Test Specimens for Copper-Base Alloy Sand Castings," Report to Subcommittee X on Copper-Base Alloy for Sand Castings, by J. J. Curran, J. W. Bolton, F. L. Wolf, and W. E. Martin, *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 199 (1942).

[3] See Editorial Note, p. 167.

No action has been taken on the adoption as standard of the Tentative Classification of Cast Copper-Base Alloys (B 119 – 40 T).

Subcommittee XI on Methods of Test for Copper and Copper Alloys (A. J. Phillips, chairman) has had two types of problems for study: one, the hydrogen or embrittlement test for copper, the other the mercurous nitrate test in several specifications where it has not previously been applied. The studies and action on these matters have not been completed.

No action has been taken to adopt as standard the Tentative Method of Test for Expansion (Pin Test) of Copper and Copper-Alloy Tubing (B 153 – 41 T) and the Tentative Method of Mercurous Nitrate Test for Copper and Copper Alloys (B 154 – 41 T).

Subcommittee XII on Publication of General Information (C. S. Cole, chairman) has been of service during the year to the chairmen of subcommittees and to the editorial staff at Headquarters in editing revisions of specifications and emergency specifications. This work applied particularly to the sections on "basis of purchase" in the specifications. The chairman has also checked a number of galley proofs of specifications connected with the publication of the 1942 issue of the compilation of A.S.T.M. Standards on Copper and Copper Alloys, sponsored by Committee B-5.

Subcommittee XIII on Tolerances (J. E. McGraw, chairman) has held three meetings with representatives of the Copper and Brass Research Assn. and as a result all of the tolerance schedules submitted to the subcommittee have been approved. At the suggestion of Chairman Greenall, the subcommittee is now reviewing all specifications under the jurisdiction of Committee B-5 looking toward bringing all tolerance tables into accord with those of the Copper and Brass Research Assn. Tolerance Standards.

This report has been submitted to letter ballot of the committee which consists of 100 voting members, 67 members returned their ballots, of whom 58 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

C. H. Greenall,
Chairman.

C. H. Davis,
Secretary.

Editorial Note

Subsequent to the Annual Meeting, Committee B-5 presented to the Society through Committee E-10 on Standards the following recommendations under the emergency procedure:

Emergency Alternate Provisions in:
Standard Specifications for Steam or Valve Bronze Castings (EA – B 61),

Additional Emergency Alternate Provisions in:
Standard Specifications for Castings of the Alloy: Copper 88 per cent, Tin 8 per cent, Zinc 4 per cent (EA – B 60a),
Standard Specifications for Composition Brass or Ounce Metal Castings (EA – B 62a),
Tentative Specifications for Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (EA – B 22a),
Tentative Specifications for Tin-Bronze and Leaded Tin-Bronze Sand Castings (EA – B 143a),
Tentative Specifications for High-Leaded Tin-Bronze Sand Castings (EA – B 144a),
Tentative Specifications for Leaded Red Brass and Leaded Semi Red Brass Sand Castings (EA – B 145a), and
Tentative Specifications for Leaded Yellow Brass Sand Castings for General Purposes (EA – B 146a).

These recommendations were accepted by Committee E-10 on December 27, 1943. The emergency provisions were published in ASTM Bulletin, No. 126, January, 1944, p. 46, and have been issued in the form of pink stickers for attachment to the specifications to which they apply.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards and tentative standards covering copper and copper alloys which are referred to earlier in this report.[1] The standards and tentative standards appear in their present form in the 1942 Book of A.S.T.M. Standards, Part I.

REVISIONS OF TENTATIVE STANDARDS

The committee recommends that the following eight tentative specifications be revised as indicated below and continued as tentative.

Tentative Specifications for Brass Sheet and Strip (B 36 – 42 T):

Table V.—Change the heading of the first part of this table reading "Slit Metal and Rolled-Edge Metal" to read "Slit Metal and Slit Metal with Rolled Edges;" also add to Table V the tolerances for rolled edge metal not previously slit as shown in the accompanying Table I.

TABLE I.—ROLLED EDGE METAL (NOT PREVIOUSLY SLIT).

Width, in.	Width Tolerances,[a] plus and minus, in.		
	0.032 in. and under in Thickness	Over 0.032 to 0.125 in., incl., in Thickness	Over 0.125 in., in Thickness
⅛ and under	0.002	0.002	.....
Over ⅛ to ⅜, incl.	0.003	0.003	0.004
Over ⅜ to 1, incl.	0.004	0.004	0.006
Over 1 to 2, incl.	0.005	0.007	0.010
Over 2 to 4, incl.	.....	0.015	0.015
Over 4 to 6, incl.	.....	0.020	0.020
Over 6	.....	0.025	0.025

[a] When tolerances are specified as all plus or all minus, double the above values.

Section 12.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and figures and the omission of the words in brackets:

> 12. (*a*) Tension tests shall be made on each lot of 5000 lb. or fraction thereof. The test specimens shall be taken parallel to the direction of rolling in such numbers and from such locations in each lot of material under test as to be representative of the quality, and not less than [five specimens] *one specimen per 1000 lb. or fraction thereof* shall be considered representative.

Change Paragraph (*b*) to read as follows by the omission of the word in brackets:

> (*b*) The number of hardness tests shall be not less than three on each of the [five] representative specimens.

Section 16.—Change from its present form: namely,

> 16. The edgewise curvature (depth (height) of arc) in a 72-in. length of metal shall not exceed the following:
>
> Flats...................... 0.5 in.
> Coils...................... 1 in.

to read as follows:

> 16. The straightness tolerances shall be as prescribed in Table VI (the accompanying Table II).

Section 17.—Change to read as follows by the addition of the italicized words and the omission of the sentence in brackets; also by the addition of a new Table VII (the accompanying Table III):

> 17. *Length Tolerances.*—The material shall be furnished in coils or flat sheet or strip, as

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[1] See p. 164.

specified. Coils shall consist of not more than three lengths, no one of which shall be less than 10 ft. in length. *The length tolerances shall be as prescribed in Table VII* (the accompanying Table III). [When furnished in coils, flat sheets or strips cut to length, a tolerance of plus 0.250 in. and minus 0.000 in. for each 10 ft. of length or fraction thereof up to 50 ft. will be permitted from the specified length.]

TABLE II.—STRAIGHTNESS TOLERANCES, IN.
Maximum Edgewise Curvature (depth (height) of arc) in any 72 in. Portion of the Total Length

Width, in.	Slit Metal		Rolled Edge Metal (Not Previously Slit)	
	As Slit	With Rolled Edges or as Straightened		
	Shipped in Rolls	Shipped Flat or in Rolls	Shipped in Straight Lengths, in Rolls, or on Bucks	Shipped on Reels or in Stagger Wound Rolls
½ and under	3	½	½	1
Over ½ to 1, incl.	1¼	½	½	1
Over 1 to 2, incl.	¾	½	⅜	..
Over 2 to 5, incl.	⅝	½	⅜	..
Over 5	½	½	⅜	..

SQUARE SHEARED METAL
Not applicable to metal over 120 in. in length

Thickness, in.	Straightness Tolerances, in.	
	Up to 10 in., incl., in Width	Over 10 in. in Width
⅛ and under	1/16	1/32
Over ⅛ to 3/16, incl.	⅛	3/64
Over 3/16	⅛	1/16

SAWED METAL
Not applicable to metal over 144 in. in length

Width, in.	Straightness Tolerances, in.
3 and under	1/16
Over 3	3/64

Section 18 (a).—Replace the present table of requirements for acceptable short lengths by a new Table VIII (the accompanying Table IV):

Tentative Specifications for Seamless Copper Tubes (B 75 - 41 T):

Section 2.—Add a new item (*3*) to read as follows, renumbering the subsequent items accordingly:

> (*3*) Electrical or nonelectrical conductors (Section 10),

Section 4.—Change the requirement for copper content of type D from "99.90 min." to read "99.92 min." per cent.

TABLE III.—LENGTH TOLERANCES, IN.

Specific Length, ft.	Automatic Machine Cut	Resquared Metal, or Square Sheared Metal
10 and under	⅛	1/16
Over 10 to 20, incl.	¼	..
Over 20	tolerances not established	..

TABLE IV.—STOCK LENGTHS WITH SHORT LENGTHS INCLUDED AND MILL LENGTHS.
Schedule of Short Lengths

Stock Length or Mill Length, ft.	Minimum Percentage by Weight of Indicated Lengths	Minimum Length of Shortest Piece, ft.	Maximum Percentage by Weight of Indicated Shortest Length
10 and 12	60	4	10
8	70	3	10
6	80	2	10

NOTE.—Length tolerances for stock lengths or mill lengths—½ in.

Tentative Specifications for Leaded Brass Sheet and Strip (B 121 - 42 T):

Make the same changes in these specifications as are recommended above in Specifications B 36.

Tentative Specifications for Cartridge Brass Cartridge Case Cups (B 129 - 42 T):

Section 4.—Change the requirement for lead content from "0.050 max." to read "0.070 max." per cent.

Note 2.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

NOTE 2.—The presence of bismuth *or mercury* in cartridge brass is objectionable and if *either is* found by the purchaser in excess of [0.006 per cent may constitute cause for rejection of the lot] *the following limits, the material shall be subject to rejection:*

Bismuth, max., per cent	*0.006*
Mercury, max., per cent	*0.0001*

Section 7.—Delete the area designated "Abnormal Zone" from Fig. 1; also delete from Table II the requirements for "Base Normal Area," and change this section to read as follows by the omission of the words in brackets:

7. Grain size measurements shall be made on at least 20 cups of type I [in an abnormal zone in the base of the cups and] in a normal zone in the side wall as indicated in Fig. 1. When viewed at a magnification of 75×, the average grain size of selected areas 79.8 mm. in diameter [in the abnormal zone in the cup base as well as] in the normal zone in the cup wall shall be in accordance with the values prescribed in Table II.

The intent of this action is to remove all requirements for grain size examination in the base of the cups and restrict the examination to the side wall.

Tentative Specifications for Gilding Metal Sheet and Strip (B 130 – 42 T):

Section 4.—Change the requirement for lead content from "0.030 max." to read "0.050 max." per cent.

Note 2.—Make the same change in this note as is recommended above in Note 2 of Specifications B 129.

Section 15.—Delete the present table of requirements for width tolerances and add the requirements as given in Table V of Specifications B 36 revised as indicated above, omitting the requirements for square sheared metal over 20 in. in width as well as the requirements for sawed metal.

Section 16.—Make the same changes in the straightness tolerances as is recommended above in Section 16 of Specifications B 36, omitting the requirements for sawed metal.

Section 17 (a).—After the second sentence, insert the following sentence and the schedule for short lengths as given in the accompanying Table IV:

When finished in stock lengths with short lengths included, and mill lengths, the schedule prescribed in Table IV shall apply.

Tentative Specifications for Gilding Metal Bullet Jacket Cups (B 131 – 42 T):

Section 4.—Change the requirement for lead content from "0.030 max." to read "0.050 max." per cent.

Note 2.—Make the same change in this note as is recommended above in Note 2 of Specification B 129.

Section 8.—In the table of grain size requirements, delete the column entitled "On Base" since it is the intent not to have any grain size requirements in these specifications for the base of the cup. In the column entitled "On Sidewall," insert the words "no requirements" opposite Class I.

Tentative Specifications for Miscellaneous Brass Tubes (B 135 – 42 T):

Section 4.—Add a note to read as follows:

NOTE.—In the case of alloy No. 3, on tube sizes greater than 5 in. in outside diameter, it is permissible to furnish tubes with a lead content less than 0.30 per cent.

Tentative Specifications for Phosphor Bronze Wire (B 159 – 42 T):

Section 1 (b).—Add the following note at the end of this paragraph:

NOTE.—Grade A spring wire is frequently subject to embrittlement due to aging. This condition is generally difficult to detect by visual means or by the standard mercurous nitrate test. To correct this condition, the wire should be subjected to a stress-relieving anneal of 6 hr. at 375 ±10 F. or its equivalent immediately prior to use.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption, revisions in three specifications, as indicated below, and accordingly asks for a nine-tenths affirmative vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society.

Standard Specifications for Rolled Copper-Alloy Bearing and Expansion Plates for Bridge and Other Structural Uses (B 100 – 40):

Section 3 (a).—Add a reference to the Tentative Specifications for Electrolytic Cathode Copper (B 115 – 41 T) as suitable for use in the manufacture of materials covered by these specifications.

Standard Specifications for Phosphor Bronze Sheet and Strip (B 103 – 42):

Section 2.—Delete item "(*3*) Grain size of annealed tempers (Section 11)," renumbering the subsequent items accordingly.

Section 3 (a).—Add a reference to the Tentative Specifications for Electrolytic Cathode Copper (B 115 – 41 T) as suitable for use in the manufacture of materials covered by these specifications.

Sections 7 and *8*.—Renumber the present Section 7 as Section 8, and change the present Section 8 to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> [8]. *7.* Phosphor bronze sheet and strip is not generally supplied in the annealed condition, therefore it is [unnecessary] *not necessary* to specify a number of anneals. [The diameter of average grain of annealed sheet and strip may be agreed upon by the manufacturer and the purchaser, and the hardness and tensile strength shall be kept within the limits prescribed in Table II.] *Grain size shall be the standard test for all thicknesses and the diameter of average grain may be agreed upon by the manufacturer and the purchaser. Rockwell hardness and tensile strength requirements shown in Table II should be considered as approximate and informatory only.*

Section 11.—Delete this section on grain size of annealed tempers which reads as follows, renumbering the subsequent sections accordingly:

> 11. Grain size shall be the standard test for phosphor bronze sheet and strip of all thicknesses in annealed tempers. Grain size limits may be agreed upon by the manufacturer and the purchaser, but in determining grain size, reference should be made to the Tentative Methods of Preparation of Micrographs of Metals and Alloys (A.S.T.M. Designation: E 2) of the American Society for Testing Materials.

Standard Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 42):

Section 3 (a).—Add a reference to the Tentative Specifications for Electrolytic Cathode Copper (B 115 – 41 T) as suitable for use in the manufacture of materials covered by these specifications.

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

Tentative Specifications for Copper Pipe, Standard Sizes (B 42 – 41 T):

The committee recommends that these specifications, revised as follows, be approved for reference to letter ballot of the Society for adoption as standard:

Section 2.—Add a new item (1) reading as follows: "(*1*) Temper (Section 4)," and renumber the subsequent items accordingly.

Tentative Specifications for Seamless Copper Tubing, Bright and Annealed (B 68 – 42 T):

The committee recommends that these specifications, revised as follows, be approved for reference to letter ballot of the Society for adoption as standard:

Section 4.—Change the requirement for copper content of type D from "99.90 min." to read "99.92 min." per cent.

Tentative Specifications for Manganese Bronze Rods, Bars, and Shapes (B 138 – 41 T):

The committee recommends that these specifications be approved for reference to letter ballot of the Society for adoption as standard without revision.

Tentative Specifications for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 – 41 T):

The committee recommends that these specifications be approved for reference to letter ballot of the Society for adoption as standard without revision.

REPORT OF COMMITTEE B-6

ON

DIE-CAST METALS AND ALLOYS

Committee B-6 on Die-Cast Metals and Alloys held two meetings during the year: one in Atlantic City, N. J., on June 25, 1942, and one in Philadelphia, Pa., on January 20, 1943, with an attendance of 29 and 18, respectively.

A balance of $84.30 was reported by Society Headquarters in the fund of Committee B-6 as of December 31, 1942. However, the committee is actually in debt to the extent of $332.00 because a bill for $416.30, incurred in the construction of exposure racks, is still unpaid.

A special committee has undertaken to raise $2000 to pay this bill and to have cash available to cover the expenses when the exposure tests come due. The committee acknowledges the receipt of $1000 from the American Die Casting Institute and is now seeking a like amount from consumers and producers who are not members of the Institute.

The following additions and changes in membership have been approved:

Howard Scott, representing American Society for Metals,
C. S. Cole (Conservation Division, War Production Board),
W. W. Hunlock, representing Gerity-Adrian Mfg. Corp.,
A. E. Martin, representing Precision Castings Co., replacing F. A. Porter (resigned),
J. Obermayer, representing Pressure Castings, Inc.,
William Romanoff (H. Kramer & Co.),
J. A. Zapf, representing Schultz Die Casting Co.,
Lt. Col. C. H. Greenall, representing Ordnance Dept., U. S. Army, Frankford Arsenal, replacing Lt. Col. L. S. Fletcher (resigned).
J. F. Ednie, representing Duquesne Smelting Corp.,
W. W. Sieg (Titan Metal Mfg. Co.),
G. L. Cherry, representing Western Electric Co., Inc., replacing C. T. Prendergast (resigned),
H. W. Schmidt, as additional representative of Dow Chemical Co.,
C. H. Hack, as additional representative of National Lead Co.,
H. W. Luetkemeyer, as additional representative of Society of Automotive Engineers,
R. W. E. Moore, as additional representative of National Electric Products Corp.,
D. L. Colwell (Conservation Division, War Production Board),
E. I. Valyi, (Lucius Pitkin, Inc.),
A. Sugar (U. S. Metals Refining Co.),

At the present time, the committee consists of 73 members, of whom 31 are classified as producers, 19 as consumers, and 23 as general interest members.

Recommendations Accepted by Committee E-10 on Standards

Subsequent to the 1942 annual meeting, Committee B-6 presented to the Society through Committee E-10 on Standards the following proposed specifications:

New Tentative Specifications for:

Copper-Base (Brass) Alloy Die Castings (B 176 – 42 T), and
Special Grade Zinc-Base Alloy Die Castings (B 186 – 42 T).

These recommendations were accepted[1] by Committee E-10 on November 27, 1942, and October 28, 1942, respectively and assigned the A.S.T.M. serial designations indicated above.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee B-6 reported the following results of the letter ballot vote from a committee membership of 66: B 176 – 42 T, affirmative 22, negative 1, ballots marked "not voting" 23; B 186 – 42 T, affirmative 30, negative 1, ballots marked "not voting" 14.

Specifications B 176 cover commercial die castings made from two brass alloys, designated as class A and class B. Class A covers copper-zinc alloys over a wide range of compositions and the copper content is limited only to a minimum of 57.0 per cent and the zinc content to a minimum of 30.0 per cent. Class B is a copper-silicon-zinc alloy having a nominal composition of 65 per cent copper, 1 per cent silicon, remainder zinc.

Specifications B 186 cover two special grade zinc-base alloy die castings for highly critical applications where, in the opinion of the purchaser, added assurance will be gained against breakdown in service which might entail danger to life or failure to achieve important objectives. The two alloys are designated as Nos. XXXIII and XXXV and correspond in type to alloys Nos. XXIII and XXV of the Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 – 41 T), respectively.

To decrease the probability of failure in service, the chemical requirements for alloys Nos. XXXIII and XXXV are prescribed within narrower limits and with lower combined impurity limits than for the regular alloys Nos. XXIII and XXV. In addition, the specifications call for routine and systematic analytical and X-ray control to insure sound castings for any entire lot of castings purchased.

Committee B-6 also submitted the following emergency recommendations to Committee E-10:

New Emergency Specifications for:

Controlled Quality Aluminum-Base Alloy Pressure-Molded Castings (ES – 29),[2]

Revision of Emergency Alternate Provisions in:

Standard Specifications for Aluminum-Base Alloy Die Castings (EA – B 85a),

Tentative Specifications for Zinc-Base Alloy Die Castings (EA – B 86a).[2]

The emergency specifications and EA – B 85a were accepted by the Standards Committee on April 15, 1943, and EA – B 86a was accepted on December 31, 1942. The emergency standard was assigned the designation indicated above and issued as a separate reprint. The revisions of the emergency alternate provisions were published in the ASTM BULLETIN, Nos. 119 and 122, December, 1942, and May, 1943, and have been published in the form of stickers for attachment to the specifications.

Emergency Specifications ES – 29 apply to all aluminum pressure-molded castings for applications where breakdown would mean serious interruption of important operations, danger to life, or failure to consummate critical objectives.

These specifications are primarily inspection and control requirements and, among other things, call for routine and systematic analytical and X-ray control.

Emergency Provisions EA – B 85a provide for the use of alloys Nos. LXXIX-A and LXXIX-B as alternates for alloys Nos. VII-A and VII-B, respectively. The alternate alloys differ mostly from the standard alloys in that the maximum silicon content has been increased from 5.5 to 9.5 per cent. However, in addition, there are a few minor revisions in the impurity contents to provide workable limits with the raw materials now allotted for die casting.

Revised Emergency Provisions EA – B86a differ from the original Emergency Alternate EA – B86 in that the maximum copper limit for alloys Nos. XXIII and XXIV was increased from 0.10 to 0.25 per cent to provide workable ranges with the raw materials now available for zinc alloy die casting.[2]

I. ADOPTION OF TENTATIVE STANDARD AS STANDARD

The committee recommends that the

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[2] See Editorial Note, p. 176.

Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 – 41 T)[3] be approved for reference to letter ballot of the Society for adoption as standard.

This recommendation has been submitted to letter ballot of the committee which consists of 73 voting members; 56 members returned their ballots, of whom 42 have voted affirmatively, 0 negatively, and 14 members marked their ballots "not voting."

II. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

The committee recommends that the following tentative specifications, which have stood for two or more years without revision, be continued as tentative without change:

Tentative Specifications for:

Magnesium-Base Alloy Die Castings (B 94 – 40 T), and

Lead- and Tin-Base Alloy Die Castings (B 102 – 39 T).

These recommendations were approved by letter ballot vote as follows: From a membership of 73 voting members, 56 members returned their ballots, 32 of whom voted affirmatively, 0 negatively, and 24 members marked their ballots "not voting" with respect to Specifications B 94 – 40 T, and 26 members voted affirmatively, 0 negatively, and 30 members marked their ballots "not voting" with respect to Specifications B 102 – 39 T.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum-Base Die Casting Alloys (E. E. Thum, chairman) prepared the Emergency Specifications for Controlled Quality Aluminum-Base Alloy Pressure-Molded Castings (ES – 29)[2] and Emergency Alternate Provisions EA – B 85a in the Standard Specifications for Aluminum-Base Alloy Die Castings (B 85 – 42), described earlier in this report.

Plans for the study of alloy No. 101(G2) (8 per cent magnesium, remainder aluminum) have been resumed and a parallel investigation was planned to be undertaken simultaneously on a 9 to 10 per cent silicon, 0.5 per cent magnesium, remainder aluminum, alloy No. 102 (SG2). Two companies have volunteered to furnish specimens of both alloys and three other companies have agreed to make tension tests. In addition, it is planned to schedule accelerated and normal aging tests on the silicon-containing alloy because of the unknown aging effects of the magnesium content. This alloy is especially recommended to replace the 13 per cent silicon alloy because of its freedom from flakes.

Subcommittee I has agreed to table the following problems now under its jurisdiction: (*1*) the study of the mechanical properties of cold chamber cast alloy No. LXXIX-B in which the iron content was to be varied from 0.6 to 2.0 per cent in 0.2 per cent increments; (*2*) the study of the influence of impurities, such as chromium and magnesium, in post war scrap; (*3*) the study of the effects of minor alloying elements on aluminum alloys. The latter problem was a joint undertaking with Subcommittee II on Aluminum Alloy Castings, of Committee B-7 on Light Metals and Alloys.

Subcommittee II on Zinc-Base Die-Casting Alloys (C. T. Prendergast, chairman) prepared the new Tentative Specifications for Special Grade Zinc-Base Alloy Die Castings (B 186 – 42 T) and the Emergency Alternate Provisions EA – B 86a in the Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 – 41 T), described earlier in this report and is now recommending that Tentative Specifications B 86 – 41 T be adopted as standard.

Subcommittee II, through a subgroup,

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[3] 1942 Book of A.S.T.M. Standards, Part I, p. 1425.

plans to prepare an article for the ASTM BULLETIN and the technical press emphasizing the need for strict adherence to the chemical requirements of the A.S.T.M. specifications for zinc-base alloy die castings.

Due to the fact that Mr. Prendergast has transferred his activities from die casting to other work at the Western Electric Co., he presented his resignation from Committee B-6. His resignation was accepted with regret.

Mr. Townsend appointed A. E. Weiss as the new chairman of this subcommittee.

Subcommittee III on Tin- and Lead-Base Die-Casting Alloys (G. O. Hiers, chairman) has not undertaken any additional work and has nothing new to report on the tests now in progress.

Subcommittee IV on Physical Tests of Die Castings (R. L. Templin, chairman) has collected data on the tests now used by the industry in determining the general satisfaction of die castings. The results of this survey were covered in an article on "Questionnaire—Tests on Die Castings," appearing in the ASTM BULLETIN, No. 118, October, 1942.

Subcommittee V on Exposure and Corrosion Tests (W. M. Peirce, chairman) has not undertaken any new exposure tests and none of the bars now in process of aging will be returned for testing prior to 1944.

Subcommittee VII on Magnesium-Base Die-Casting Alloys (J. A. Gann, chairman) still has under consideration a proposed revision of the Tentative Specifications for Magnesium-Base Alloy Die Castings (B 94 – 40 T) to incorporate requirements for a new alloy to be known as alloy No. 13X. However, the subcommittee has not yet released information on this subject to the committee for approval.

Subcommittee VIII on Copper-Base (Brass) Die-Casting Alloys (J. C. Fox, chairman) has been organized to investigate and write specifications for copper-base alloys.

This subcommittee prepared the new Tentative Specifications for Copper-Base (Brass) Alloy Die Castings (B 176 – 42 T), described earlier in this report.

This report has been submitted to letter ballot of the committee which consists of 73 voting members; 54 members returned their ballots, of whom 51 have noted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

G. L. WERLEY,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-6 presented to the Society through Committee E-10 on Standards a proposed revision of the Emergency Specifications for Controlled Quality Aluminum-Base Alloy Pressure Molded Castings (ES – 29). The revised emergency specifications were accepted by Committee E-10 on November 24, 1943, and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 146.

On January 8, 1944, Committee E-10 accepted proposed revisions in the Emergency Alternate Provisions in the Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 – 43). The revised emergency provisions, bearing the designation EA – B 86b, were published in ASTM BULLETIN, No. 126, January, 1944, p. 46, and have been issued in the form of a pink sticker for attachment to the standard.

REPORT OF COMMITTEE B-7

ON

LIGHT METALS AND ALLOYS, CAST AND WROUGHT

Committee B-7 on Light Metals and Alloys, Cast and Wrought, has held two meetings during the past year: one in Atlantic City, N. J., on June 24, 1942, and the other in Buffalo, N. Y., on March 3, 1943. Several of the subcommittees held meetings during this period.

Since the 1942 annual meeting, Committee B-7 has gained four new members. The membership of the committee now totals 52, of whom 22 are classified as producers, 19 as consumers, and 11 as general interest members.

A number of new subcommittee appointments have likewise been made during the year. R. W. Waring has replaced C. H. Greenall as chairman of Subcommittee II on Aluminum Alloy Castings, Lt. Col. Greenall having asked that he be relieved of this responsibility. A new Subcommittee VII on Codification of Light Alloys has been organized, under the chairmanship of I. V. Williams, to maintain the new nomenclature system adopted during the year.

As in the previous year, the activities of this committee have been somewhat handicapped by the added pressure on its membership resulting from the war emergency. The uncertainty with respect to the availability and allocation of certain materials still hampers many of the changes indicated as desirable in certain of the specifications. The necessity for using the maximum amount of secondary or low-grade materials for many products has also introduced specification problems which are rather difficult to answer to date. Although some of the specifications under the jurisdiction of this committee should normally be considered for adoption as standard at this time, this uncertainty has made it desirable to withhold such action. The committee is, therefore, recommending that many of these tentative standards be continued as tentative pending better stabilization of materials and product requirements.

Of particular interest in connection with the work of this committee is the progress report of Subcommittee VI on Anodic Oxidation of Aluminum and Aluminum Alloys,[1] covering the preliminary results of cooperative tests comparing the performance of anodic coatings in the salt spray test with their behavior under atmospheric conditions. It is hoped further to expand the testing program of this subcommittee to include an investigation of the relative resistance to corrosion of anodic coatings applied to various aluminum-base casting alloys.

A program has also been started by Subcommittee II to study the methods of casting tension test specimens of the aluminum-base alloys. A working subgroup has been established for this purpose and for developing a recommended design of test bar gating.

I. NEW TENTATIVE STANDARD

The committee recommends that the Specifications for Aluminum Sheet and Plate for Use in Welded Pressure Vessels[2] be accepted for publication as tentative. These specifications were prepared by Subcommittee III at the request of the Boiler Code Committee of The American Society of Mechanical Engineers.

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[1] See p. 182.

[2] These specifications were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 242.

II. Revisions of Tentative Standards

The committee recommends that the following six tentative specifications be revised as indicated below and continued as tentative. These revisions are desired in the case of the specifications for wrought alloys to bring them in line with present commercial products as produced by the newer manufacturing methods. In the case of the specifications for casting alloys, the changes are desired to provide improved castings or greater availability of material.

Tentative Specifications for Aluminum Sheet and Plate (B 25 – 42 T):[3]

Section 3.—Add the following to the table of requirements as to chemical composition:

Total of all elements except aluminium, max., per cent	1.0

Table I.—Change the requirements for elongation of half-hard temper sheet and the accompanying footnote to read as follows by the addition of the italicized figures and the omission of the words and figures in brackets:

Thickness, in.	Elongation in 2 in., min., per cent
0.013 to 0.019	[3] *2*[a]
0.020 to 0.031	[4] *3*[a]
0.032 to 0.050	[5] *4*[a]
0.051 to 0.113	[7] *5*[a]
0.114 to 0.249	7

[a] [This value is required for flat sheet. For coiled sheet in the half-hard temper, the elongation may be less than that for flat sheet by 1 per cent in 2 in.] *The maximum thickness of coiled or strip sheet commercially available in the half-hard temper is 0.080 in.*

Tentative Specifications for Aluminum-Base Alloy Sand Castings (B 26 – 42 T):[3]

Table I.—Change the chemical requirements for alloy C2 to read as follows by the addition of the italicized figures and the omission of those in brackets:

	Alloy C2
Copper, per cent	4.5 ± 0.5
Iron, max., per cent	[1.2] *1.0*
Silicon, max., per cent	1.2
Magnesium, max., per cent	[0.10] *0.05*
Manganese, max., per cent	[0.3] *0.5*
Zinc, max., per cent	[0.2] *0.5*
Titanium, max., per cent	0.2
Total other impurities, max., per cent	[0.2] *0.4*
Aluminum	remainder

Change the silicon requirement for alloy ZG41 from "0.4 max." to read "0.25 max." per cent.

Tentative Specifications for Aluminum-Manganese Alloy Sheet and Plate (B 79 – 42 T):[3]

Table I.—Change the requirements for elongation of half-hard temper sheet and the accompanying footnote to read as follows by the addition of the italicized figures and the omission of the words and figures in brackets:

Thickness, in.	Elongation in 2 in., min., per cent
0.010 to 0.019	[3] *2*[a]
0.020 to 0.031	[4] *3*[a]
0.032 to 0.050	[5] *4*[a]
0.051 to 0.113	[6] *5*[a]
0.114 to 0.161	7
0.162 to 0.249	8

[a] [This value is required for flat sheet. For coiled sheet in the half-hard temper, the elongation may be less than that for flat sheet by 1 per cent in 2 in.] *The maximum thickness of coiled or strip sheet commercially available in the half-hard temper is 0.080 in.*

Tentative Specifications for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 41 T):[3]

Table I.—Change the chemical requirements for the following elements of alloy CS4 to read as follows by the addition of the italicized figures and the omission of those in brackets:

Manganese, max., per cent	[0.3] *0.5*
Zinc, max., per cent	[0.2] *0.5*
Total other impurities, max., per cent	[0.3] *0.4*

Tentative Specifications for Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 – 42 T):[3]

Table I.—Change the requirements for elongation of one-quarter hard and half-hard temper sheet and the accompanying footnote to read as follows by the addition of the italicized figures and

the omission of the words and figures in brackets:

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[3] 1942 Book of A.S.T.M. Standards, Part I.

Temper	Thickness, in.	Elongation in 2 in., min., per cent
One-quarter hard	0.016 to 0.019	[5] *4*[a]
	0.020 to 0.050	[6] *5*[a]
	0.051 to 0.113	[8] *7*[a]
	0.114 to 0.249	[10] *9*[a]
Half hard	0.010 to 0.019	[4] *3*[a]
	0.020 to 0.050	[5] *4*[a]
	0.051 to 0.113	[7] *6*[a]
	0.114 to 0.249	[8] *7*[a]

[a] [This value is required for flat sheet; for coiled sheet in the one-quarter hard and half-hard tempers, the value may be less than that for flat sheet by 1 per cent in 2 in.] *The maximum thickness of coiled sheet commercially available in the one-quarter hard temper is 0.102 in., and in the half-hard temper is 0.060 in.*

Tentative Specifications for Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (**B 126 – 42 T**):[3]

Table I.—Change the requirements for elongation of quarter-hard temper sheet and the accompanying footnote to read as follows by the addition of the italicized figures and the omission of the words and figures in brackets:

Thickness, in.	Elongation in 2 in., min., per cent
0.016 to 0.019	[4] *3*[a]
0.020 to 0.031	[5] *4*[a]
0.032 to 0.050	[6] *5*[a]
0.051 to 0.113	[7] *6*[a]
0.114 to 0.161	8
0.162 to 0.249	9
0.250 to 2.000	8

[a] [This value is required for flat sheet. For strip sheet in the quarter-hard temper, the elongation may be less than that for flat sheet by 1 per cent in 2 in.] *Coiled or strip sheet is available commercially in the thicknesses of 0.016 to 0.102 in.*

III. Tentative Standards Continued as Tentative Without Change

In accordance with Society Regulations, the committee has reviewed those tentative standards which have stood for two years without revision and recommends that the following specifications and methods of test be continued as tentative without change:

Tentative Specifications for:

Aluminum Ingots for Remelting (B 24 – 41 T),
Magnesium-Base Alloy Sand Castings (B 80 – 41 T),
Magnesium-Base Alloy Sheet (B 90 – 41 T),
Magnesium-Base Alloy Forgings (B 91 – 41 T),
Magnesium-Base Alloys in Ingot Form for Sand Castings and Die Castings (B 93 – 41 T),
Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 41 T),
Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings (B 112 – 41 T)

Methods of Test for:

Dielectric Strength of Anodized Aluminum (B 110 – 39 T),
Sealing of Anodically Coated Aluminum (B 136 – 40 T), and
Weight of Coating on Anodically Coated Aluminum (B 137 – 40 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 52 members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE

Items	Affirmative	Negative	Ballot Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Spec. for Aluminum Sheet and Plate for Use in Welded Pressure Vessels	31	0	11
II. REVISIONS OF TENATIVE STANDARDS			
Spec. for Aluminum Sheet and Plate (B 25 – 42 T)	31	0	11
Spec. for Aluminum-Base Alloy Castings (B 26 – 42 T)	32	0	10
Spec. for Aluminum-Manganese Alloy Sheet and Plate (B 79 – 42 T)	29	0	13
Spec. for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 41 T)	34	0	8
Spec. for Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 – 42 T)	27	0	15
Spec. for Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 – 42 T)	27	0	15

Activities of Subcommittees

Subcommittee I on Aluminum and Aluminum Alloy Ingots (A. Sugar, chairman).—This subcommittee has reported no activity during the past year.

Subcommittee II on Aluminum Alloy Castings (R. W. Waring, chairman).—Revisions in several specifications under the jurisdiction of this subcommittee have been prepared, as mentioned earlier in this report. Members of this subcommittee at the March meeting agreed to offer their services in the development of information or data on an unofficial basis. It was felt that considerable information of value in the war effort could be made available more expediently in this way than through normal committee channels. The chairman has offered to act as a clearing agent for requests for such data.

A study of test bar mold design for sand-cast aluminum-base tension test specimens has been initiated by the committee, a subgroup having been set up to carry on the necessary work. It is hoped that this study will ultimately result in submittal to the Society of a standard test bar gating design.

Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys (P. V. Faragher, chairman).—This subcommittee has prepared, at the request of the A. S. M. E. Boiler Code Committee, the proposed Specifications for Aluminum Sheet and Plate for Use in Welded Pressure Vessels which are being recommended for publication as tentative, as mentioned earlier in this report.

In addition, several changes in elongation requirements for sheet have been recommended in connection with other tentative specifications under its jurisdiction, details of which are given earlier in this report. With the use of modern high-speed rolling equipment for the production of flat sheet, which product provides the same properties as now specified for coiled sheet, such changes are indicated to bring these specifications in line with present commercial products.

Subcommittee IV on Magnesium and Magnesium Alloys, Cast and Wrought (J. A. Gann, chairman).—This subcommittee has reported no activity during the past year. A review of the various specifications under its jurisdiction has resulted in the recommendation that they be continued as tentative without change.

Subcommittee V on Testing of Light Alloys (R. L. Templin, chairman).—This subcommittee has reviewed the question of developing a standard sand-cast test bar mold design and casting procedure and agreed that this problem should basically be handled by Subcommittee II. Should questions of design of specimen or methods of testing develop as part of this problem, subcommittee V will be glad to cooperate.

Subcommittee VI on Anodic Oxidation of Aluminum and Aluminum Alloys (J. D. Edwards, chairman).—A progress report covering the preliminary results of cooperative tests comparing the performance of anodic coatings when exposed to salt spray and to the weather has been prepared by this subcommittee and is appended hereto.[1] It is planned to continue the weather exposure tests for an additional period before attempting to make a final comparison of the results. The subcommittee has also had under consideration a test program to investigate the relative resistance to corrosion of anodic coatings applied to various aluminum-base casting alloys.

Subcommittee VII on Codification of Light Alloys (I. V. Williams, chairman).—This subcommittee is reviewing at the present time a code system for the magnesium-base alloys and expects shortly to submit recommendations to Committee B-7. The code system adopted last year for the aluminum-base alloys has been submitted to Committee B-6 on Die-Cast Metals and Alloys for consideration in connection with its specifications for aluminum die castings.

This report has been submitted to letter ballot of the committee which consists of 52 members; 42 members returned their ballots, of whom 34 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

SAM TOUR,
Chairman.

H. J. ROWE,
Secretary.

APPENDIX

A COMPARISON OF THE PERFORMANCE OF ANODIC COATINGS ON WROUGHT ALUMINUM ALLOYS WHEN EXPOSED TO SALT SPRAY AND TO THE WEATHER

Prepared by Junius D. Edwards[1]

Tests to compare the performance of anodic coatings in the salt spray[2] with their behavior under atmospheric exposure conditions have been started by four cooperating laboratories. A description of the test panels is given in Table I.

In the salt spray tests, specimens Nos. 2, 3, 4, 5, 6, and 7 showed the greatest resistance to attack, as evidenced by the summary given in Table II. In the examination of these specimens, the presence of a few small pinholes, which did not increase appreciably with continued exposure, was not considered evidence of attack. In one laboratory, there was no attack on these particular specimens after 6 months exposure, and in another laboratory the same condition existed after 700 hr. exposure, at which time the tests were discontinued. In the third laboratory, there was no attack after 2100 hr. exposure, with the exception of specimen No. 4, which showed some attack after 1100 hr. in the salt spray. These tests are quite consistent in indicating good resistance to attack by these six specimens for periods of 700 hr. or more exposure to the standard salt spray conditions.

TABLE I.—DESCRIPTION OF TEST PANELS FOR A.S.T.M. EXPOSURE TESTS.

Specimen	Alloy Coated	Electrolyte	Sealing Procedure	Time of Coating, min.	Thickness of Coating, in.	Weight of Coating, mg. per sq. in.
No. 2.............	52S	Sulfuric acid	Hot water	20	0.00028	12.6
No. 3.............	52S	Sulfuric acid	Hot water	30	0.00039	16.0
No. 4.............	52S	Sulfuric acid	Dichromate	10	0.00012	4.9
No. 5.............	52S	Sulfuric acid	Dichromate	20	0.00029	11.6
No. 6.............	52S	Sulfuric acid	Dichromate	30	0.00037	16.3
No. 7.............	17S-T	Sulfuric acid	Dichromate	30	0.00041	12.3
No. 8.............	17S-T	Chromic acid	No seal	30	0.00005	1.5
No. 9.............	17S-T	Sulfuric acid	Hot water	30	0.00033	9.7

The results of salt spray tests at a fourth laboratory were quite at variance with those just reported. The attack commenced sooner and, in general, was more severe. For example, some attack on specimens Nos. 4, 5, 6, and 8 was reported after 160 hr. in the salt spray and on all specimens after 400 hr. exposure. In looking for the reason for this pronounced variation in behavior, it was found by spectrographic examination that the surface of these samples was heavily contaminated with copper and nickel, and with minor amounts of lead and tin. Presumably this contamination came from brass, bronze, and other metal specimens which had been tested with the same salt solution. This is a condition which should be avoided in salt spray testing of bare or oxide-coated aluminum. While these results, therefore, are not comparable with the other tests, they are included in Table II because they illustrate the necessity of using salt solutions free from heavy metals.

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[1] Assistant Director of Research, Aluminum Company of America, New Kensington, Pa.

[2] In accordance with Tentative Method of Salt Spray Testing of Non-Ferrous Metals (B 117 - 41 T), 1942 Book of A.S.T.M. Standards, Part I, p. 1469.

TABLE II.—RESULTS OF SALT SPRAY TESTS.

NOTE.—The rating "none" includes those panels showing only a few small pinhole points of attack.

Specimen	Laboratory Reports First Attack			
	Laboratory A	Laboratory B	Laboratory C	Laboratory D
No. 2	None[a]	None[b]	None[c]	300
No. 3	None[a]	None[b]	None[c]	400
No. 4	None[a]	1100 hr.	None[c]	160
No. 5	None[a]	None[b]	None[c]	160
No. 6	None[a]	None[b]	None[a]	160
No. 7	None[a]	None[b]	None[c]	300
No. 8	600 hr.	200 hr.	200 hr.	160
No. 9	600 hr.	700 hr.	200 hr.	300

[a] After 6 months exposure.
[b] After 2100 hr. (about 3 months) exposure.
[c] After 700 hr. exposure.

In line with other experience, the oxide coatings on alloy 17S-T tended to be less protective than on alloy 52S, which is inherently more resistant to corrosion.[3] Moreover, the coating on panel No. 8, even though made according to a standard specification, seemed to be somewhat thinner than the average of coatings of this type. One of the cooperating laboratories, therefore, prepared another panel of 17S-T coated for 60 min. in chromic acid electrolyte; this panel had a coating thickness of 0.00020 in. and a coating weight of 4 mg. per sq. in. In the salt spray test it showed an initial breakdown after 200 to 300 hr. exposure. Another panel prepared by the same laboratory, but anodically coated for 120 min., showed no evidence of failure after 2100 hr. exposure to salt spray.

These tests, however, were not intended to develop the relative merits of various coating methods but only to correlate their performance in the salt spray test with their behavior during atmospheric exposure. It was perhaps fortunate, therefore, that at least two of the panels showed relatively early failure in the salt spray tests. The results, generally speaking, indicate that while salt spray exposure tests may discover coatings subject to early failure, still the resistance of most anodic coatings to this type of exposure is sufficiently extended so that the method could scarcely be considered an accelerated test.

Atmospheric exposures at four locations (Chicago, Philadelphia, Miami, and New Kensington) revealed no definite attack on any of the specimens after 6 months. In the New Kensington exposure, definite evidence of attack had developed on panel No. 8 after 10 months. At Point Judith, R. I., close to the sea, after 6 months exposure, specimens Nos. 4, 8, and 9 showed some evidence of attack, while at Oakland, Calif., after the same period, all of the specimens except No. 3 showed definite evidence of attack. At Chicago, after 13 months exposure, panels Nos. 2, 3, 5, 6, 7, 8, and 9 showed very slight pitting, whereas panel No. 4 showed appreciable attack. The atmospheric tests will be continued for longer periods before attempting a final comparison with the results of the salt spray test.

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[3] Nominal compositions: alloy 17S, copper 4.0 per cent, manganese 0.5 per cent, magnesium 0.5 per cent; alloy 52S, magnesium 2.5 per cent, chromium 0.25 per cent; aluminum and normal impurities constitute the remainder of each alloy.

REPORT OF COMMITTEE B-8

ON

ELECTRODEPOSITED METALLIC COATINGS

Committee B-8 on Electrodeposited Metallic Coatings held one meeting during the year, on June 25, 1942, in Atlantic City, N. J. Activities since that time have been carried on by correspondence and letter ballot.

One resignation from the committee, that of G. F. Jenks has been received and accepted with much regret. Nineteen new members have been added during the year. The membership now totals 69, of whom 27 are classified as producers, 8 as consumers, and 34 as general interest members.

The activities of the committee are largely the result of suggestions and requests made by Governmental agencies, and are directed toward the furthering of the war effort.

Subsequent to the 1942 annual meeting' Committee B-8 presented to the Society through Committee E-10 on Standards a new Recommended Practice for Chromium Plating on Steel for Engineering Use. This recommended practice was accepted[1] by Committee E-10 for publication as tentative on March 6, 1943, and is available in separate reprint form under the designation B 177 - 43 T.

The committee also submitted to Committee E-10 proposed Emergency Specifications for Electrodeposited Coatings of Lead on Steel, described under the activities of Subcommittee I. This recommendation was accepted by Committee E-10 on May 29, 1943, and the emergency standard issued as a separate reprint under the designation ES - 31.

I. New Tentative Standard

The committee recommends that the new Recommended Practice for Preparation of Low-Carbon Steel for Electroplating[2] be accepted for publication as tentative.

II. Tentative Standards Continued as Tentative

The committee recommends that the following tentative specifications and methods of test be continued as tentative without revision:

Tentative Specifications for:

Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 - 41 T),

Electrodeposited Coatings of Nickel and Chromium on Copper and Copper-Base Alloys (B 141 - 41 T),

Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys (B 142 - 41 T), and

Tentative Methods of Test for:

Local Thickness of Electrodeposited Coatings (A 219 - 40 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 69 members, with the results shown in Table I.

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee B-8 reported results of the letter ballot vote as follows: Of a total membership of 67, 63 members returned their ballots, of whom 50 voted affirmatively, 0 negatively, and 13 members marked their ballots "not voting."

[2] This recommended practice was accepted as tentative by the Society and appears in the 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 322.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Rec. Practice for Preparation of Low-Carbon Steel for Electroplating	40	2	9
II. TENTATIVE STANDARDS CONTINUED AS TENTATIVE			
Spec. for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 – 41 T)	41	1	18
Spec. for Electrodeposited Coatings of Nickel and Chromium on Copper and Copper-Base Alloys (B 141 – 41 T)	42	1	17
Spec. for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys (B 142 – 41 T)	40	1	19
Test for Local Thickness of Electrodeposited Coatings (A 219 – 40 T)	43	1	16

The committee has sponsored the following four papers which are being presented at this annual meeting of the Society:

- "Properties of Plated Lead Coatings on Steel," by Gustaf Soderberg, The Udylite Corp.[3]
- "Chromium Plate in Engineering Applications—Its Thickness and Finishing," by T. G. Coyle, United Chromium, Inc.[4]
- "Use and Misuse of the Salt Spray Test as Applied to Electrodeposited Metallic Finishes," by C. H. Sample, Bell Telephone Labs., Inc.[5]
- "Factors Underlying Specifications for Electrodeposited Coatings," by E. M. Baker, University of Michigan.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications and Definitions (Gustaf Soderberg, chairman) has been active through its subgroups.

The Subgroup on Lead Coatings (T. H. Westby, chairman) prepared the Emergency Specifications for Electrodeposited Coatings of Lead on Steel (ES – 31). According to information received, preliminary thin copper coatings proved advantageous in accelerated corrosion tests. However, since no outdoor exposure tests were available to substantiate this, types of lead coatings have been provided in the specifications both with and without such copper strike coatings. The paper on "Properties of Plated Lead Coatings on Steel," by the chairman of the subcommittee, being presented at this annual meeting,[3] was the basis for the thicknesses specified in the emergency standard.

The Subgroup on Tin Coatings (H. A. Pray, chairman) has found no demand for specifications at this time.

The Subgroup on Silver Coatings (A. K. Graham, chairman) has not been active.

The Subgroup on Definitions (William Blum, chairman) has postponed its work for the duration of the war.

Subcommittee II on Performance Tests (L. R. Westbrook, chairman) reluctantly had to accept the resignation of its original chairman, C. E. Heussner, because of illness. This subcommittee has not yet become active.

Subcommittee III on Conformance Tests (C. H. Sample, chairman) has conducted a questionnaire on the salt spray test, a summary of which appears in the Appendix. The chairman of the subcommittee has made a survey of the use and misuse of salt spray testing of electrodeposited coatings which is being presented in a paper at this annual meeting.[5]

Subcommittee IV on Electroplating Practice (A. K. Graham, chairman) has been active through two subgroups.

The Subgroup on Cleaning Practice (M. M. Beckwith, chairman) prepared the Recommended Practice for Preparation of Low-Carbon Steel for Electroplating being recommended for publication as tentative.

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[3] See p. 562.
[4] See p. 556.
[5] Published in ASTM BULLETIN, No. 123, August, 1943, p. 19.
[6] See p. 191.

The Subgroup on Chromium Plating (R. M. Wick, chairman) prepared the Tentative Recommended Practice for Chromium Plating on Steel for Engineering Use (B 177 - 43 T). The paper by T. G. Coyle on "Chromium Plate in Engineering Applications—Its Thickness and Finishing," being presented at this annual meeting[4] provides additional information relating to specific applications.

Subcommittee V on Supplementary Protective Finishes for Metallic Coatings (B. H. McGar, chairman) met in June, 1942, in Atlantic City, N. J., and discussed in particular the possibilities of preparing specifications for supplementary coatings on electroplated zinc and cadmium, which field had been released by Committee A-5 on Corrosion of Iron and Steel to Committee B-8. The conclusion was reached that sufficient data have not yet been developed, but that the subcommittee members should watch the field closely and report to the chairman.

This subcommittee has assisted in giving advice on subjects within its sphere, when requested by those engaged in war work.

This report has been submitted to letter ballot of the committee which consists of 69 members; 51 members returned their ballots, of whom 49 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. M. BAKER,
Chairman.

GUSTAF SODERBERG,
Secretary.

RESOLUTION ON DEATH OF E. M. BAKER

The following Resolution on the death of E. M. Baker, chairman of Committee B-8, was presented to the Society at the annual meeting:

Whereas our dear friend and highly esteemed chairman, Edwin Myron Baker, passed on to his rewards on May 27, 1943, and

Whereas Committee B-8 of the American Society for Testing Materials at its annual meeting assembled wishes to express to Mrs. Baker and other members of his family its gratitude for his skillful guidance of the work of the committee from its inception and his outstanding contribution to electrochemical engineering, be it hereby,

Resolved, that the members and officers of Committee B-8 of the American Society for Testing Materials express personally and collectively to Mrs. Baker and other members of Professor Baker's family their very great sorrow at the loss of a dear friend, colleague and guiding spirit; hoping, however, that the fact that he will always live in blessed memory and through his great contributions to his chosen field of endeavor, will in some measure be a comfort in their bereavement.

June 29, 1943.

E. A. ANDERSON,
Vice-Chairman.

GUSTAF SODERBERG,
Secretary.

REPORT OF SUBCOMMITTEE III ON CONFORMANCE TESTS

Report of Replies to Questionnaire on Salt Spray Testing

At the meeting of Committee B-8, held at the Society's Annual Meeting in June, 1942, Subcommittee III was instructed to review the status of the salt spray test in this committee and prepare an authoritative statement regarding its use and limitations. In accordance with this instruction, a questionnaire was submitted to the members of Committee B-8 which at that time included seventy-five individual and corporate members. Thirty-three (44 per cent) replies to the questionnaire were received and are summarized and generalized below under headings corresponding to the questions asked. Very few actual data were received in support of the opinions expressed regarding the value of the test and the correlation of its results with the service performance of various protective coatings. In this respect the replies to the questionnaire were somewhat disappointing; however, the wide divergence of opinions expressed and the apparent lack of available data are important indications of a need for more experimental evidence on which the value and limitations of the test, as it is applied to the many possible coating-basis metal combinations, may be definitely established. The questionnaire is appended for the convenience and interest of readers of this report.

Use of Salt Spray:

Replies to the questionnaire indicate that the salt spray test is used by 27 out of the 33 persons who answered. Of these, 15 employ the test for checking conformance with outside specifications but only two of the 15 report satisfactory correlation of the results with actual service tests. A helpful observation concerning the six persons who *do not* use the salt spray test, but who have been good enough to acknowledge the questionnaire, is found in four statements of disapproval of the test as it now exists, either because of lack of correlation with other better controlled tests and service performance or because of lack of data establishing the value of the test. Considerable use of the test for revealing the porosity of electropositive type coatings such as copper, nickel, etc., is also made. The test is also used as an indirect measure of the thickness of electronegative coatings such as zinc and cadmium.

Materials Tested:

From the replies to the questionnaire, it is apparent that the salt spray test is being used for all the common protective coating-basis metal combinations currently employed in industry. In addition to electroplated finishes this includes paints, lacquers and other organic coatings, such as oils and greases on ferrous and non-ferrous metals, oxide and phosphate coatings on steel and anodic coatings on aluminum.

Operation of Salt Spray Test:

The construction, capacity and operation of salt spray equipment in current use vary considerably. Sizes vary from a few cubic feet capacity to the dimensions of a small room. Variations in size, however, are not important as such, provided the environmental conditions within the space are the same. Salt solutions currently used in the boxes are, for the most part, 20 per cent sodium chloride. Also employed to a limited extent for the test are 10 per cent sodium chloride and $3\frac{1}{2}$ per cent sodium chloride solutions, as well as natural and synthetic sea water.

A temperature of 95 F. (35 C.) has been maintained by eleven users in preference to "room temperature" which is used by twelve of those answering. Other operating temperatures reported range from 85 to 150 F. (30 to 65 C.).

In general, care is exercised to atomize only fresh clean solution and to discard that which might be contaminated with corrosion products[1] in accordance with accepted salt spray testing practice.

Air pressures employed to atomize the solution vary from 8 to 80 psi. There are several instances of unmeasured air pressure reported.

The position of the test specimens within the box varies from the horizontal to the vertical. In this connection it is noteworthy that several expressed the opinion that it is important to so place the specimen that all sides and surfaces are uniformly exposed to the spray. On several occasions it has been observed that vertically suspended test panels were not uniformly wetted by the spray even after several days exposure. Obviously this is a possible source of considerable variability in test results and it would appear from the number of persons employing different positions of test specimens, that further agreement and standardization might be reached on this significant and controllable variable.

Fog density is, in practically all cases, unmeasured or described simply as "dense." One report indicates that density of fog was controlled by maintaining a spray sufficiently dense to obscure the details of a 100-watt lamp at a distance of five feet.

Exposure periods employed vary from 1 to 500 hr. depending in some instances on the requirements of specifications, or in other instances on the particular purpose for which the test is being performed. In most cases the definition of the end point of the test is ambiguous varying from the presence of "any corrosion" within a specified time to "total failure." In this connection it should be emphasized that if salt spray test results are to be used in specifications as a basis for acceptance or rejection the end point requirements or just what constitutes failure should be clearly defined.

Reproducibility of results in the same box is reported as favorable or satisfactory in the majority of cases. In four cases it was reported as fair while in four others it was considered poor. Reproducibility in other boxes is, in most cases, unknown due to lack of comparative tests in different boxes.

Comparison with Service and Outdoor Tests:

The incomplete information received concerning correlation of salt spray with service and outdoor tests is mostly negative in character. Of 24 users of the salt spray test, five report good correlation, eight report that they have "no data" on this point, or that data are insufficient to draw conclusions. Poor or unsatisfactory correlation is specifically reported by eleven persons. One of these submitted experimental evidence showing negative correlation between the time for initial failure of nickel *versus* nickel-chromium coatings on steel in the salt spray test and the behavior of these coatings exposed to an outdoor industrial atmosphere. These data, however, showed good correlation when the protective value of nickel coatings of different thicknesses were compared. A former user of the salt spray test discontinued its use because of erratic results. Mention was made of the necessity for establishing a ratio of salt spray life to exposure life for each coating-basis metal combination. Such studies should prove valuable because judging from the brief reports mentioning specific metals there is considerable disagreement in regard to the relation between salt spray and actual service behavior.

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[1] In one notable instance, not reported in reply to the questionnaire, this is not done. In this case the solution is atomized (on alternate three minute cycles) by the impingement of a jet on a rapidly rotating disk and after drenching the test specimens the solution falls to the bottom of the monel metal test chamber which is maintained at 55 C. by means of immersion heaters. It is then pumped through metal tubes back to the atomizer to repeat the cycle for 100 hr. after which it is discarded. While this test may not be considered as a salt spray test by its users, considerable equipment being manufactured for the Armed Forces is required to withstand it. Design engineers unfamiliar with salt spray testing refer to the test merely as a salt spray test and generally confuse it with the more widely used standard test procedure such as described in A.S.T.M. Tentative Method of Salt Spray Testing of Non-Ferrous Metals, (B 117 - 41 T), 1942 Book of A.S.T.M. Standards, Part I, p. 1469. The test is mentioned in this report primarily in the interest of completeness and in an attempt to avoid future confusion.

Comparison with Other Tests:

Very few data were received in regard to the comparison of salt spray and other porosity tests used. Other porosity tests currently employed include the ferroxyl test, revised ferroxyl test, hot water test, humidity tests, micrographic examinations, electrographic tests, and corrosive vapor tests. As indicated previously in this report most users of the salt spray test use it for testing the porosity of protective coatings. However, indications are that comparison of salt spray and other porosity tests are not entirely satisfactory.

Other tests used for comparing the quality of coatings include tests for adherence, thickness measurements, seawater immersion tests, alternate hot and cold water shock tests and the Preece test, as well as outdoor exposure and service tests in various atmospheres. While there seems to be general agreement that an accelerated test for comparing the quality of protective coatings is needed, there is also an expression of the feeling that the salt spray test is not, in its present form of application, adequate for use as the only accelerated test to be employed for this purpose.

Limitations and Value of the Test:

The principal limitations of the test given in the replies include lack of correlation with outdoor exposure, destruction of the specimen, poor reproducibility, and lack of agreement in interpretation of results. For the most part opinions in regard to the value of the test as currently employed are stated qualitatively and inconclusively. Statements to the effect that the test is a good comparative indication of the general quality level of coatings of the same type, but should not be used as sole basis for making quality evaluations were contributed by six persons. The test is used for its "value in indicating comparative porosity" by seven observers, three value the test for indicating particularly "inferior coatings," five persons use the test for "rating several coatings in the order of their superiority." The speed of the test in relation to other tests available is listed as an important advantage by a few users.

Summary:

Considerable variations exist in current salt spray testing techniques in spite of the fact that acceptable standard procedures such as A.S.T.M. Tentative Method B117 - 41 T and Government Specification AN-QQ-S-91 are generally available. From the replies it is apparent that uniform control of operating temperatures, position of test specimens, fog density and air pressure is not accomplished by most users. It should be pointed out, however, that variations are possible even though the test is performed in accordance with A.S.T.M. Tentative Method B 117 – 41 T. Thus it would appear that further standardization and agreement may be necessary to insure satisfactory reproducibility among different laboratories.

Opinions vary widely in regard to the suitability of the salt spray test for measuring the quality of electrodeposited metallic coatings and there is an apparent lack of experimental evidence correlating salt spray and service behavior of the many possible coating-basis metal combinations. This is particularly true in regard to the degree of correlation between salt spray tests made at different times in different places. In spite of its shortcomings, the salt spray test is widely used in specifications as a basis for acceptance or rejection of electroplated products and is regarded by many as the best accelerated test available for revealing particularly inferior protective coatings. Granting that the test has many advantages in this respect, the conclusion often drawn that coatings which will withstand 200 hr. exposure to this test are satisfactory, while coatings which will not are unsuitable, irrespective of the type of coatings or the basis metals to which they are applied, is misleading.

Respectfully submitted on behalf of the subcommittee,

C. H. SAMPLE,
Chairman.

SALT SPRAY TESTING QUESTIONNAIRE

1. Do you use a salt spray test, and if you do, for what purpose?
   (*a*) Adherence to outside specifications.
   (*b*) Testing of porosity.
   (*c*) Comparison of different coatings.
2. What coating—basis metal combinations are you testing in the salt spray?
3. How do you operate your salt spray test?
   (*a*) Size of box.
   (*b*) Solution composition.
   (*c*) Temperature and where measured.
   (*d*) Air pressure.
   (*e*) Position of test pieces.
   (*f*) Fog density.
   (*g*) Exposure periods.
   (*h*) End point constituting failure for different combinations of coatings and basis metals.
   (*i*) Reproducibility in your box.
   (*j*) How well do you reproduce behavior in other boxes?
4. (*a*) If the answer to No. 1 (*b*) is yes, what other tests have you used for porosity testing?
   (*b*) How do these tests compare with the salt spray for different combinations of coatings and basis metals?
5. If the answer to No. 1 (*c*) is yes, what other tests have you used to compare different coatings?
6. How well does the salt spray test correlate with service tests and outdoor exposure tests for different combinations of coatings and basis metals?
7. How well do the other tests which you have used compare with service and outdoor exposure tests?
8 What in your opinion is the value and limitations of the salt spray test?

Any data which you can submit in support of your answers to Nos. 6, 7, and 8 above will be of value.

APPENDIX

FACTORS UNDERLYING SPECIFICATIONS FOR ELECTRODEPOSITED METALLIC COATINGS

By Edwin M. Baker[1]

The war has created many emergency situations not the least of which has been the necessity of writing specifications without adequate time or opportunity to investigate fundamentals and to gather data which should properly form the basis of these specifications. This has been true in many fields, including that of electrodeposited metallic coatings applied to war equipment for the purpose of protecting base metals under various conditions of service. In many instances no data are available concerning the thickness and other characteristics of the deposit which determine the resistance to corrosion under the conditions to be met in service, which may be world-wide. Examples of such new uses of plating are zinc-plated pennies, zinc- or copper-plated steel cartridge cases, chromium-plated steel mess trays, and plated steel communication equipment to be used in tropical jungles. The writing of specifications for these and other examples should be facilitated by both existing and newly proposed A.S.T.M. specifications on electrodeposited metallic coatings, even though these may not be based on the new service conditions. In view of this situation, it has been suggested that this would be a proper time to discuss the fundamental factors which have underlain specifications sponsored by Committee B-8 on Electrodeposited Metallic Coatings, and which might apply to all specifications in this field.

Coating Thickness Most Important

It is to be noted that in specifications such as A.S.T.M. Tentative Specifications for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 – 41 T),[2] Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys (B 142 – 41 T),[3] and Electrodeposited Coatings of Nickel and Chromium on Copper and Copper-Base Alloys (B 141 – 41 T),[4] the essence of these specifications is the minimum thickness of the electrodeposited metallic coating or coatings on significant surfaces. Specifications A 166 and B 142 also provide for a continuity test which has taken the form of a salt spray test.

It should be emphasized here, as in the specification, that the quality of the plate is specified primarily by thickness, and that the salt spray requirements are sufficiently lenient that they become effective only if the plate has not been applied under properly controlled conditions from baths of proper composition. Hence, rejection or acceptance will normally hinge on the thickness test and not upon the results of the salt spray test. The salt spray test then becomes effective as a means of rejection only when the conditions under which plating is carried out are decidedly defective or improper. This may not be apparent from a simple reading of the specifications, and would not be true if the required times in the salt spray without failure were greatly increased. The desired relationship between the functions of the thickness and salt spray tests is achieved by specifying a salt spray test for a smaller number of hours than are *normal for good deposits* of the respective thicknesses.

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[1] Chairman, Committee B-8 on Electrodeposited Metallic Coatings; Department of Chemical Engineering, University of Michigan, Ann Arbor, Mich.

[2] 1942 Book of A.S.T.M. Standards ,Part I, p. 1454.

[3] *Ibid*, p. 1461.

[4] *Ibid*, p. 1458.

Correlation of Thickness and Exposure Tests:

The above distinction is a proper one and has a fundamental basis in that there is a correlation between the outdoor exposure or weather resistance and the thickness of the electrodeposited metallic coatings. In contrast, there has never been established a satisfactory correlation between the results of the salt spray test and the results of weather exposure tests, except perhaps in certain seacoast localities where weather exposure is primarily exposure to salt-laden atmosphere, and even then the correlation is not all that might be desired.

Over a period of more than 15 yr., research has been carried out at the National Bureau of Standards under a cooperative program with the American Electro-Platers' Society on the preparation and testing of panels of iron, copper, zinc, and their alloys, plated with various sequences and thicknesses of copper, nickel, and chromium. These panels have been exposed upon the racks of the American Society for Testing Materials at Pittsburgh, New York, N. Y., Sandy Hook, Key West, Washington, D. C., and State College, Pa. The results of these tests have appeared in the publications of the American Electro-Platers' Society and of the National Bureau of Standards.[5]

These test results already published will not be discussed in detail in this paper, and at this point it is desired merely to call attention to the fact that while numerous accelerated tests have been made on these coatings, the only basis upon which any reasonable correlation could be made was upon the thicknesses of the electrodeposited coatings.

Minimum Thickness Important:

All of the specifications referred to above contain paragraphs relative to the minimum thickness of each of the electrodeposits in a sequence, or of the total minimum thickness, upon significant surfaces. Significant surfaces in the case of decorative plating are defined as those surfaces that are visible and subjected to wear or corrosion or both. In the case of much equipment manufactured today, it is not necessary that the surface be visible in order to be significant. Significant surfaces are obviously those which are of importance in the use of the plated object.

Average Thickness Not Specified:

It is to be noted that one will not find in the specifications a requirement as to the average thickness of plate. If failure of the part occurs in service, failure would be expected where the plate had the least thickness, provided this area was one which was exposed to normal mechanical wear or corrosion or both. It is of no value to have an article, part of which would resist wear or corrosion or both for 5 yr. if other important parts of the same article fail from one or both of these causes in 6 months. Upon articles of complicated shape it is admitted that it may be more difficult to produce electrodeposited coatings to meet a certain specification of minimum thickness than it is to meet a specification requiring a certain average thickness. On the other hand, the purpose of the specifications is to insure, so far as insurance may be obtained by specifications, that the article will give certain satisfaction in service. The prime purpose of the specifications is not, and should not be, for ease in manufacturing. Ease of manufacture is a proper objective of the original design of the article.

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[5] William Blum, Paul W. C. Strausser and Abner Brenner, "Protective Value of Nickel and Chromium Plating on Steel," *Journal of Research*, Nat. Bureau of Standards, Vol. 13, No. 3, September, 1934, p. 331 (*Research Paper 712*).

Paul W. C. Strausser, Abner Brenner, and William Blum, "Accelerated Tests of Nickel and Chromium Plating on Steel," *Journal of Research*, Nat. Bureau Standards, Vol. 13, No. 4, October, 1934, p. 519 (*Research Paper 724*).

William Blum and P. W. C. Strausser, "Outdoor Exposure Tests of Electroplated Nickel and Chromium Coatings on Steel and Nonferrous Metals," *Journal of Research*, Nat. Bureau Standards, Vol. 24, No. 4, April, 1940, p. 443 (*Research Paper 1293*).

Lux, *Proceedings*, Am. Electro-platers' Soc., p. 43 (1941).

Lux and Berdick, *Proceedings*, Am. Electro-platers' Soc., p. 19 (1942).

SALT SPRAY TEST ON CATHODIC COATINGS

In the specifications thus far considered, the electrodeposited metallic coatings are cathodic to the base metal and, therefore, protect only in so far as they are impervious or, possibly, in so far as pores in the coating are so small that they become quickly and effectively closed with corrosion product. The protection afforded by the coating will, therefore, be first of all a function of the initial porosity of the coating and later a function of the porosity of the coating at that later stage in the life of the article. "Incipient" pores, which are present in the coating at the time of manufacture in the form of minute areas covered by relatively thin coatings, may develop at some later date into existent pores, as the result either of mechanically wearing away of a portion of the coating or as the result of the corrosion or weathering away of a portion of the coating. This is one of the reasons why a test for initial porosity is not *in and of itself* a satisfactory test for the resistance to corrosion under service conditions which the plated article must meet, even though initial porosity is important. Many researches on the protective value of electrodeposited coatings which are cathodic to the base metal have shown that the porosity decreases under any given plating condition with increasing thickness of the coating. Therefore, from both the standpoint of the initial porosity and of the porosity that will exist after some period in service, the thickness of the coating must fundamentally be a good measure of the protection that it will afford against corrosion. If only one characteristic of the deposit could be specified, thickness would seem to be the most important.

The salt spray test does have an application in the case of cathodic coatings in that it will discover and make apparent pores which either exist in the coating or which may conceivably be created as the result of the amount of corrosion of the coating which may take place during the salt spray test. This is its prime purpose in Committee B-8 specifications, where it appears as a secondary test, that is, a test for "continuity of coating." The salt spray test is not regarded by Committee B-8 as a *complete* measure of the quality of cathodic coatings. For deposits of *equal thickness*, the salt spray test does distinguish as to quality. Hence, while it is a secondary test, it has an important function in these specifications.[6] An accelerated test that would give results correlating with actual performance on weather exposure is desired but is not available. Thus far, the salt spray test is our best accepted measure of continuity of cathodic coatings.

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[6] For a further discussion of the salt spray test see Report of Subcommittee III on Conformance Tests, of Committee B-8, p. 187; also C. H. Sample, "Use and Misuse of the Salt Spray Test as Applied to Electrodeposited Metallic Finishes," published in ASTM BULLETIN, No. 123, August, 1943, p. 19.

ANODIC COATINGS—ZINC AND CADMIUM

While anodic electrodeposited coatings, such as zinc and cadmium over steel, do not come within the province of Committee B-8, it may not be out of order to point out that much the same considerations apply. The big difference is that for such coatings the salt spray test is not a measure of initial porosity. The salt spray is an even poorer means of approximating the thickness of such coatings, and consequently does not appear at all in specifications covering coatings of zinc or cadmium over steel.

While an anodic coating will protect an exposed base metal over considerable distances, such as $\frac{1}{8}$ or $\frac{1}{16}$ in., under certain conditions, as in the presence of a strong electrolyte, it should not be assumed that porosity in such coatings is unimportant. It is fundamental that such protection may be achieved only at the cost of a somewhat more rapid consumption of the protecting plate in the areas adjacent to the exposed base metal.

NEW SPECIFICATIONS FOR LEAD COATINGS

The proposed new Emergency Specifications for Electrodeposited Coatings of Lead on Steel[7] are based on both minimum thickness and salt spray tests, although there is indication that under some exposure conditions lead coatings may perform as weakly anodic coatings.[8]

NEW RECOMMENDED PRACTICE SPECIFICATIONS

Committee B-8 is proposing for acceptance in June new tentative standard specifications of a different type, namely, Recommended Practice for Preparation of Low-Carbon Steel for Plating.[7] This is of the same type as the new Tentative Recommended Practice for Chromium Plating of Steel for Engineering Use (B 177 - 43 T).[7] Both of these recommended practices set forth procedures which have been carefully examined in detail and which are known to give satisfactory results if followed faithfully. It is recognized that it is not legally sound to specify both a procedure for manufacturing a product and also the result to be obtained. These specifications delineate the procedure but not the result. They may be properly combined with result specifications which delineate requirements on coating thickness or salt spray test results or both. While electroplating is not an overnight development, it is, nevertheless, a young industry in many ways, and there is ample evidence of a real need for these two new recommended practices. In the short time interval between submission of B 177 to Committee B-8 and the approval by the Society's Committee E-10 on Standards, there came to the attention of the author two definite examples where the application of the recommended practice, in place of procedures formerly used, resulted in satisfactory products in place of failure to obtain acceptable products.

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[7] 1943 Supplement to Book of A.S.T.M. Standards, Part I, pp. 150, 322, and 327.

[8] Gustaf Soderberg, "Properties of Plated Lead Coatings on Steel," see p. 562.

REPORT OF COMMITTEE C-1

ON

CEMENT

Because of the war emergency, Committee C-1 on Cement held only one meeting during the year instead of the usual two. The committee met in March at Allentown, Pa., which location was very convenient for many of the members. The meeting was well attended.

The Emergency Alternate Specifications for Portland Cement (EA – C 150), issued in 1942, have continued in force unchanged. However, it has been reported that certain cement, which had been ground close to the lower fineness limit of that emergency alternate, had exhibited excessive "bleeding" when used in concrete. The special advisory subcommittee which was appointed last year, under the chairmanship of J. L. Miner, to consider matters relating to W.P.B. limitations on cement, has been instructed to investigate this bleeding and its relation to fineness and make such recommendations as may seem appropriate.

SUBCOMMITTEE ACTIVITIES

The volume of subcommittee work was necessarily less than in the preceding year but, even under the emergency conditions, considerable progress was reported by the subcommittees.

Chemical Analysis:

The Working Committee on Methods of Chemical Analysis, under the chairmanship of W. C. Hanna, has continued its studies of those chemical determinations which have been of particular interest in the recent work of Committee C-1. Much attention was given to the determination of Vinsol resin in portland cement. The Hercules Powder Co. had developed a method for the determination of Vinsol resin in portland cement by means of the determination of the methoxyl content. Six laboratories participated in the subcommittee's study of that method and of certain modifications thereof as proposed by others, including the suggestions from C. E. Wuerpel, T. A. Hicks, and F. P. Diener. That study is being continued on samples of cement with known Vinsol resin content, some of the samples containing known amounts of mineral lubricating oil or tallow along with the resin.

The procedures for determining certain constituents in cement as described in Sections 1 to 13 of the Tentative Methods of Chemical Analysis of Portland Cement (C 114 – 42 T) have been published for some years and could well be considered for adoption as standard. However, these tentative methods have just been published in the 1942 Book of A.S.T.M. Standards and the subcommittee thought that such consideration should be deferred for a time because of the considerable number of editorial changes which would be required to incorporate these sections of Tentative Methods C 114 – 42 T in Standard Methods C 114 – 42. Therefore, Committee C-1 recommends that Tentative Methods C 114 – 42 T be continued as tentative for another year without change.

Volume Change and Soundness:

As a result of recent considerations, the Working Committee on Volume Change and Soundness (H. F. Gonnerman, chairman) recommended to Committee C-1 for adoption as standard the Tentative Method of Test for Autoclave Expansion of Portland Cement (C 151 – 40 T)[1] with the following changes:

Section 2 (d).—Change the third and fourth sentences to read as follows by the addition of the italicized figures and the omission of those in brackets:

> The automatic pressure control shall be capable of maintaining the pressure at 295 ± [5] *10* psi. for at least 3 hr. A gage pressure of 295 ± [5] *10* psi. corresponds to a temperature of [215.7 ± 1 C. (420.3 ± 2 F.)] *215.7 ± 1.7 C. (420.3 ± 3 F.).*

Section 2 (e).—Change the second and third sentences to read as follows by the addition of the italicized words and figures and the omission of the figures in brackets:

> The instrument shall be graduated to [0.0001] *at least 0.001* in., and when tested at any point throughout its range, the error shall not be greater than plus or minus [0.0004] *0.002* in. The difference between repeated measurements shall not be greater than [0.0001] *0.001* in.

Section 5 (b).—Change the third sentence to read as follows by the addition of the italicized figures and the omission of those in brackets:

> The 295 ± [5] *10* psi. pressure shall be maintained for 3 hr.

Committee C-1 has approved the proposal of the Working Committee and recommends to the Society that Tentative Method C 151 be revised as indicated above and approved for reference to letter ballot of the Society for adoption as standard.

Strength:

The Working Committee on Strength (O. L. Moore, chairman) has given consideration to numerous details of the Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 – 37 T) and has recommended approval of the proposed revision appended[2] to this report. One of the principal changes is an increase in the tolerance for distance between compartment faces of used molds for the 2-in. cubes. This change will permit the acceptance of many molds which are now in use and are considered satisfactory but fail to meet the present more stringent requirement for this dimension. Not having completed some of the studies which were undertaken in the past year or two, the Working Committee recommended that the revised Tentative Method C 109 be continued as tentative.

Committee C-1 has approved the proposal of the Working Committee and recommends to the Society that Tentative Method C 109 be revised as indicated and continued as tentative for another year.

Fineness:

The Working Committee on Fineness (P. H. Bates, chairman) offers no recommendations affecting standards this year. However, the subcommittee has been active and some of the members have engaged in cooperative studies of the possibilities of the air-permeability method as a rapid and convenient test for the fineness of portland cement. Modified air-permeability methods for determining the fineness of cement have been developed by Messrs. Rigden, of the Road Research Laboratory (England); Boehler, of the Universal Atlas Cement Co.; and Blaine, of the National Bureau of Standards. In each of these methods, the time required for some definite volume of air to pass through a bed of powder is determined. The test method is rapid and the fineness values obtained with any one instrument are reproducible. Ten "Blaine Fineness Meters," which are very inexpensive and are easily made and operated, have been distributed to various laboratories for comparative tests.

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[1] 1942 Book of A.S.T.M. Standards, Part II, p. 1077.

[2] This revised method was accepted as tentative by the Society and appears in the 1943 Supplement to Book of A.S.T.M. Standards, Part II, p. 79.

Alkalies in Cement:

The Working Committee on the Effects of Alkalies in Portland Cement on the Durability of Concrete (R. F. Blanks, chairman) has no report to render just now. However, the subcommittee has been devoting much attention to its problem. The chairman of the subcommittee has recently submitted a paper wherein he reviews and summarizes field and laboratory work on this problem to date. Some of the subcommittee members and other interested parties have also presented discussions on this subject. Because of the interest attached to discussions of this problem, these personal contributions are appended to this report as "Notes on the Effect of Alkalies in Portland Cement on the Durability of Concrete."

Sulfate Resistance:

The Working Committee on Sulfate Resistance (D. G. Miller, chairman) is now completing a report on the comparative short-time tests of 121 commercial cements for sulfate resistance. Six laboratories cooperated in these short-time tests and the results will be received with much interest because those same cements had already been compared in long-time exposure tests for their alkali resistance.

Heat of Hydration:

The Working Committee on Heat of Hydration (G. J. Durant, chairman) has been studying details of a method for the determination of the heat of hydration of portland cement and is considering recommending it for publication as tentative. The proposed method is essentially a revision of the method now described in the Federal Methods of Testing Cement (SS-C-158a) and employs the same simple thermos jar calorimeter.

A paper is being presented at this annual meeting entitled "A Study of the Heat of Solution Procedure for Determining the Heat of Hydration of Portland Cement" by Leo Shartsis and Edwin S. Newman.[3]

Miscellaneous Subcommittee Work:

The other working committees had continued the study of their problems, accumulating data or outlining future work, but they had no conclusive reports or recommendations to make at this time. The sponsoring committees have been attentive to the details of specifications relating to their respective fields, but make no recommendations affecting standards on cement at this time.

Cement Reference Laboratory:

The Cement Reference Laboratory at the National Bureau of Standards continued to be active throughout the year under the supervision of the Subcommittee on the Cement Reference Laboratory (G. E. Warren, chairman). This joint project of the Bureau and the A.S.T.M. completed the fourteenth year of its work under the sponsorship of Committee C-1. The work of the laboratory for the period July 1, 1942, to March 31, 1943, is summarized below:

The seventh inspection tour, begun late in 1940, was completed, 266 cement laboratories having been visited since then. The early start of the eighth tour was announced and requests for inspection have already been received from 208 laboratories. More requests are confidently expected, but the total for the new tour will probably be smaller than usual because of the efforts to devote the work to those laboratories which are testing cement for Federal, State, or war emergency projects.

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[3] See p. 905.

The Public Roads Administration continued to give financial aid to the Reference Laboratory and still requires Reference Laboratory inspections at laboratories which test cement for certain projects.

Verification of compression testing machines was continued. The data collected on the many different machines have proven interesting and helpful. Many machines which were verified were being used for tests of materials other than cements. Some of the materials were of military nature and this part of the laboratory's work has thus been closely linked to the war effort.

The proving rings which the Reference Laboratory uses in the verification of compression testing machines have been checked occasionally for performance. However, some months ago a complete recalibration of all six rings was made by the Engineering Mechanics Section of the Bureau. It was very reassuring to find that the performance of the rings in the recalibrations was substantially the same as when the rings were first calibrated about two years earlier. This performance record is of more than ordinary interest since the laboratory had records showing the approximate number and magnitude of the loadings on each ring during its use in the field. The loadings had been quite uniformly distributed among the rings.

The inspectors have reported numerous changes in the staffs of some cement laboratories and it is probably on that account that in recent months there has been more than the usual need for criticism and instruction relating to test methods. Accordingly, the inspectors have increased the time devoted to the details of procedure.

Recommendations Affecting Standards:

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 78 voting members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVE STANDARD			
Test for Compressive Strength of Portland-Cement Mortars (C 109 – 37 T)	61	1	2
II. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Test for Autoclave Expansion of Portland Cement (C 151 – 40 T), as revised	64	0	4

This report has been submitted to letter ballot of the committee, which consists of 78 voting members; 69 members returned their ballots, of whom 67 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

P. H. BATES,
Chairman.

G. E. WARREN,
Secretary.

APPENDIX

NOTES ON THE "EFFECT OF ALKALIES IN PORTLAND CEMENT ON THE DURABILITY OF CONCRETE"*

This appendix to the Report of Committee C-1 on Cement presents some notes relating to the problems of the Working Committee on the "Effects of Alkalies in Portland Cement on the Durability of Concrete." This material has been contributed by the chairman of that subcommittee, by some of its members, and by others interested in the subject.

CONTRIBUTED BY R. F. BLANKS[1]

Many articles have appeared in contemporary scientific literature, related to the subject of concrete deterioration due to adverse reactions between alkalies in cements and some constituents of aggregates. An up-to-date bibliography is appended[2] for ready reference.

Instances of concrete deterioration attributed in whole or in part to adverse alkali-aggregate reactions are characterized by the following observable conditions: (1) open cracking, usually of random pattern on a fairly large scale; (2) excessive internal and over-all expansion; (3) while the cracks may be very large at the concrete surfaces (openings of up to 1½ in. have been observed), they extend into the concrete only a relatively short distance (6 to rarely more than 18 in.); (4) gelatinous exudations and whitish amorphous deposits, both on the surface and within the mass of the concrete, especially in voids and adjacent to pieces of aggregate; (5) areas of what appear to be zones of reactivity, alteration or infiltration around aggregate particles, particularly those containing opal and certain types of acid and intermediate volcanic rocks; and (6) lifeless chalky appearance of the concrete, below exposed surfaces.

The list of important concrete structures so affected continues to grow, as will be evident from the following tabulation. In all of these cases the deleterious effects of adverse chemical reactions between high-alkali cements and aggregates have been established beyond any reasonable doubt.

Highway pavement and structures	California
Buildings	California
Street paving	California
Railway bridge piers	California
Portions of Friant Dam	California
Highway pavement and structures	Washington
Highway structures	Idaho
American Falls Dam	Idaho
Parker Dam	Arizona-California
Gene Wash and Copper Basin Dams	California
Coolidge Dam	Arizona
Street paving	Kimball, Nebraska
Highway pavement and structures	Kansas
Buck Dam and power plant	Virginia
Portions of Owyhee Dam	Oregon

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* See Editorial Note, p. 220.

[1] Materials and Testing Engineer, Bureau of Reclamation, Denver, Colo., Chairman of Working Committee on the Effects of Alkalies in Portland Cement on the Durability of Concrete.

[2] See p. 219.

In several of the above-recorded cases, the particular aggregates involved have been used in other structures with low-alkali cements, and the service records have been either entirely satisfactory or at least materially improved. Service comparisons with high- and low-alkali cements have been made for paving and structures in Monterey, San Luis Obispo, Santa Barbara, Ventura, and Los Angeles counties, Calif.; highway structures in Washington; street paving at Fresno, Calif.; Parker, Gene Wash, and Copper Basin Dams, in comparison with the corresponding pumping plants; Coolidge Dam in Arizona; and Friant Dam in California. There are indications that other structures will be added to both lists when all the facts and data are collected.

Intensive investigations and tests on the effects of alkalies in cement are currently in progress at a number of laboratories. There are listed below some of the laboratories and investigators of whose work the author has direct knowledge. This list does not presume to be all-inclusive.

California State Highways, T. E. Stanton, Sacramento, Calif.
Washington State Highways, Bailey Tremper, Olympia, Wash.
California Portland Cement Co., W. C. Hanna, Colton, Calif.
University of California, R. E. Davis, Berkeley, Calif.
Bureau of Reclamation, R. F. Blanks, Denver, Colo.
Kansas State Highways, C. H. Scholer, Manhattan, Kans.
Portland Cement Association, F. R. McMillan, Chicago, Ill.
Tennessee Valley Authority, P. J. Freeman, Knoxville, Tenn.
National Bureau of Standards, P. H. Bates, Washington, D. C.
Massachusetts Institute of Technology, R. W. Carlson, Cambridge, Mass.
U. S. Engineers, C. E. Wuerpel, Mt. Vernon, N. Y.
Dewey & Almy Chemical Co., H. L. Kennedy, Cambridge, Mass.
Public Roads Administration, F. H. Jackson, Washington, D. C.

From the above list, it will be readily appreciated that large volumes of data and information are being accumulated. Many facts have been established, accompanied, as might be expected, by some seeming anomalies. The complexity of the alkali-aggregate problem is reflected by the fact that, with all the intensive study which it is receiving, practical methods for predetermining troublesome combinations of materials have not been discovered, nor have entirely satisfactory methods been evolved for correcting combinations known to be adversely reactive. In the following paragraphs an attempt will be made to list the known facts, point out some of the apparent anomalies that have been encountered, and direct attention to some phases of the problem for which answers are most acutely needed.

The rate at which expansive reaction (outwardly evidenced by excessive surface cracking) develops in concrete structures in service varies (to mention a few of the known factors) with the type of reactive aggregate involved, the alkali content of the cement, the thickness of section, the quality of the concrete, and the conditions of exposure, particularly with respect to moisture conditions within and surrounding the concrete. At Parker Dam in Arizona, severe open surface cracking developed with the advent of warm weather 2 yr. after the structure was completed. At Buck Dam excessive expansion was apparently not observed until 10 to 15 yr. after construction. In those cases where early, rapid expansion occurs, the action generally seems to slow down or even stop after a few years. The slower cases appear to be more insidious and deterioration progresses, comparatively slowly, for 30 yr. and longer, or until rehabilitation is required. Almost any combination of variations from the extreme sets of conditions may occur.

Several forms of gelatinous deposits, presumed to be directly or indirectly the products of chemical reactions between alkalies in the cement and siliceous constituents in the aggregates, have been collected from affected structures and analyzed both chemically and microscopically. Glass-like beads, ranging from clear to opaque, from gray to brown, have been found in a few cases exuding from the surfaces of concrete in the field. The beads may appear following a rainy period or where moisture is migrating through a wall or slab of concrete. White or gray amorphous deposits, varying in hardness with moisture conditions, are very common. When cores are drilled from affected concrete and stored in the presence of moisture, exudations of clear or opaque jelly-like substances appear on the surfaces.

Chemical analyses of the deposited material, whatever its form, invariably identify it as sodium and potassium silicates, although the relative proportions of alkalies, silica, and water vary, and varying amounts of other substances such as calcium may also occur. As mentioned above, these gels may be transparent but they may be white or gray, with a porcelaneous luster, an effect which is associated with an admixture of one or more crystalline phases with the amorphous phases. Calcium sulfo-aluminate and calcium carbonate are commonly found with the gel.

Petrographic examinations indicate that the gel frequently occurs in preexisting voids or pores, in cracks or joints in aggregate particles (and in the continuations of such cracks into the surrounding concrete matrix), in "sockets" of aggregate grains, or impregnating porous portions of the concrete or aggregate. It is noteworthy that these "reaction products" are commonly associated spacially in a selective manner with certain aggregate types. Although these aggregate types are presumed to be the reactive constituents of the aggregate, it is not established that their reaction with alkalies in the cement is simple; in many cases the physical or chemical changes observable in the pebbles do not seem to be quantitatively sufficient to account for the relatively large amounts of associated "reaction products," a fact that suggests contribution from some other source.

Aggregate pebbles which appear to have experienced reaction in affected concrete exhibit one or more of the following features:

(*a*) The pebbles are frequently fractured, the cracks commonly extending outward into the surrounding concrete matrix. These cracks are usually wholly or partially filled with the gelatinous or other products described above.

(*b*) The pebbles have an outer border which appears darker than the interior when viewed in hand specimen, but which is translucent and therefore lighter in color when viewed by transmitted light in thin section. Although this border-phenomenon may be the effect of chemical reaction, there is frequently so little observable difference between the physical and mineralogical character of the exterior and interior parts of the pebble that a simple infiltration of gelatinous material into the outer parts of the pebble is suggested.

(*c*) The pebbles are frequently surrounded or partially surrounded by gelatinous and associated substances which have lodged in the pebble "socket."

(*d*) The pebbles are frequently softened, discolored, or carry discolored zones around the outer surface. In moist specimens, such pebbles frequently are wet with a viscous gelatinous liquid.

Some of these criteria cannot be certainly ascribed to action within the concrete unless the observation is supplemented by a detailed study of the raw aggregate to determine its "before and after" character. It is usually not possible to ascertain definitely whether deposits filling cracks or surrounding aggregate particles have entered preexisting openings or have made the openings through the exertion of pressures consequent to their precipitation, or to some other cause.

Strength, elastic properties, and volume-change tests have been made on cores drilled from concrete in affected structures. In general such cores have developed higher strengths than might be expected from the dead, chalky or lifeless appearance of the concrete. Young's modulus values are low, being in the range of one-half, or less, the modulus for normal concretes. Length change measurements on cores stored in the presence of moisture show gradual expansions over an indefinite period of time. Corresponding expansions have been measured in the structures.

The exact mechanics by which large cracks develop at the surfaces of concrete structures, yet extend into the body of the concrete only a comparatively short distance, have not been satisfactorily explained. Apparently, the forces causing expansion are greatly reduced or eliminated in the surface concrete, either by drying (for air exposure) or by leaching (for submerged conditions). The residual mixing water in the interior concrete is sufficient to promote alkali-aggregate reactions. The interior moisture supply may, of course, be augmented from outside sources in many ways. It has been our belief from the outset that the observable evidences of deterioration are the direct result of internal expansion and that the mechanism of this expansion requires special explanation.

It has been suggested or implied at various times that expansions may result simply from the crystallization of new compounds formed by the chemical (alkali) reactions, or through the mechanisms of hydration, or that the formation of the gel might be attended by expansive tendencies. In an attempt to explore other possible mechanisms for the expansive action, experiments on the osmotic properties of concrete have been undertaken. In these experiments, neat cement and concrete wafers are set up as osmotic diaphragms between two liquids, water on one side and sodium silicate or sodium hydroxide on the other. These experiments, although incomplete, indicate that hardened cement and concrete under certain conditions can behave osmotically to cause significant pressures.

The favorite laboratory approach to the alkali problem has been a study of the expansion by means of volume-change specimens, incorporating the active aggregate in combination with both high and low-alkali cement. The elastic modulus of the same specimens, as measured by sonic methods, has also been observed by some laboratories. It has been found that, in cases where reactions have developed, the expansion is accelerated by moderately elevated temperature (100 to 130 F). The presence of moisture is essential to the development of expansion. If small specimens are immersed in water, expansion does not occur, seemingly because the alkalies are extracted from the mortar; however, expansion does develop in large specimens under such storage.

Combinations of reactive aggregate and cement in concrete or mortar bar specimens generally produce expansions in proportion to the alkali content of the cement. However, with some combinations the order of expansion at later ages is not always directly related to the alkali content. Several investigators have reported results for a particular aggregate, showing at later ages expansions that are greater with cements of moderate alkali content than with higher alkali cements, even though the high-alkali cements gave the greatest initial expansion. However, it has been found that certain proportions of active constituents in an aggregate produce expansions greater than those resulting when the active ingredients are present in greater or lesser amounts. This optimum amount of reactive aggregate has been shown to differ with the alkali content of the cement, being smaller with cements of low-alkali content and greater with high-alkali cements. Thus, apparently anomalous results may be produced by cement to aggregate proportions greater or less than the most active combinations.

Very small amounts of the extremely active components, such as opaline silica, appear to be sufficient to impart activity to an aggregate in the presence of a high-alkali cement. If present in an excessive amount no deleterious expansion occurs; for instance, opal, or the very active siliceous magnesian limestone referred to by Mr. Stanton (of which the opal content (approximately 20 per cent) has been determined apparently to be the prime if not the only reactive mineral), when used in their entirety do not expand even with a high-alkali cement.

The grain size of the reactive constituent also controls the degree of expansion with a given cement and the proportion in a mix at which it demonstrates greatest activity. Concrete bars made with a high-alkali cement and crushed quartz graded 0 to $\frac{3}{4}$ in., in which varying percentages of certain size fractions of the quartz particles were replaced by the corresponding sizes of siliceous magnesian limestone, developed greatest expansions for the following combinations: replacment in the Nos. 16 to 8 sieve size—$2\frac{1}{2}$ per cent limestone, $97\frac{1}{2}$ per cent quartz; in the Nos. 30 to 16 sieve size—5 per cent limestone, 95 per cent quartz; and in the Nos. 50 to 30 sieve size—10 per cent limestone and 90 per cent quartz. Greater or lesser replacements in the several size fractions resulted in lower expansions than attained by the above optimum combinations. Maximum expansion in mortar bars was obtained when 19 per cent of the siliceous magnesian limestone was substituted for quartz sand in the Nos. 50 to 30 sieve size range.

It has also been found that the amount of reaction is affected by the richness of the mix, the richer mixes exhibiting greater expansions and greater initial rates of reaction. The evidence so far gathered indicates that there are many factors influencing the amount of expansion produced by the contact of reactive aggregates with high-alkali cement and that a fortuitous combination of circumstances may result in considerable reaction whereas some departure therefrom can yield conflicting results. The combination which gives maximum expansion with one cement may not be the same for another.

Some experimentation has been reported on additions which might inhibit the reaction between aggregate and cement. Aluminum powder has demonstrated that it possesses value for this purpose, but must be used in an amount which produces too much hydrogen gas, lowering the unit weight and strength of the concrete. Additions of calcium chloride have been found to accelerate and increase the expansive reaction between cement and aggregate.

Pumicite additions to the concrete reduce the expansion to a fair degree, but pumicite is more effective if used to replace the high-alkali cement in the concrete mix. Mr. Stanton has shown that additions of several finely ground siliceous materials, as a replacement of cement, diminish the expansive reaction much more than do comparable reductions in the amount of high-alkali cement accomplished without the replacement. This is particularly true of opal which, however, if present in large granules, has demonstrated itself as one of the most reactive materials with high-alkali cement. It appears that the properties which make a fine-ground material desirable as a pozzuolana prohibit its use in coarser sizes as an aggregate in conjunction with high-alkali cement. It is significant in this connection that, in general, little or no expansive reaction has been obtained with reactive aggregate and portland-pozzuolana cement, whatever its alkali content.

The attached table (Table I) lists some of the aggregates that have been studied in the Bureau of Reclamation laboratories and which show expansion when combined with high-alkali cement but fail to show expansion with a low-alkali cement. Practically all of these aggregates are associated with deteriorating concrete structures exhibiting expansive cracking.

TABLE I.—EXPANSIONS SECURED WITH REACTIVE AGGREGATES IN 1:2 MORTARS.

2 by 2 by 10 in. bars cured at 100 F. in sealed storage

(Bureau of Reclamation Laboratory Tests)

Sand	Source	Age, months	Expansion, per cent		Sand[a]
			1.42 per cent Alkali in Cement	0.18 per cent Alkali in Cement	
Blue River	Kan.	16	0.084	0.006	All Blue River
Republican River	Kan.	16	0.184	0.008	All Republican River
Phyllite Buck Dam	Va.	14	0.201	−0.004	No. 8—19%
Vallecito Dam	Colo.	14	0.228	0.006	All Vallecito Dam
Pumping Plant Intake—Metroplitan Water District	Calif.	14	0.320	0.003	All M.W.D. Sand
Coolidge Dam	Ariz.	9	0.321	−0.006	All Coolidge Dam
Coolidge Dam Crushed Gravel	Ariz.	9	0.384	−0.004	All Coolidge Dam
Kimball	Nebr.	16	0.403	0.012	All Kimball
Oro Fino	Calif.	16	0.441	0.011	All Oro Fino
White River	Wash.	3	0.311	−0.004	All White River
Cowlitz River	Wash.	16	0.625	0.006	All Cowlitz River
Selected reactive material, Parker Dam aggregate	Calif.	6	0.706	−0.004	No. 100—19%
American Falls Dam	Idaho	1	0.032	−0.010	All American Falls Dam
Idaho Falls Underpass	Idaho	16	0.726	0.003	All Underpass
Siliceous magnesian limestone	Calif.	16	0.859	−0.001 (12 months)	No. 50—19%

[a] Sand composed mainly of uniformly graded crushed quartz, with active aggregate replacing the quartz in the screen size and amounts shown, unless shown as all active aggregate.

Information obtained from field observations, petrographic studies, and observations of length changes of laboratory bars combine to indicate that the most conspicuously reactive of the common aggregate materials are:

- Opal
- Other highly siliceous rocks (such as some cherts, some siliceous limestones, some chalcedonies, and intimate opal-chalcedony admixtures)
- Certain acid to intermediate volcanic rocks
- Miscellaneous types (such as Buck phyllite) which may also react because of their siliceous character.

It cannot be said that this list is complete; other types, concerning which our present data are incomplete or ambiguous, may be found to be reactive to a minor degree or under special circumstances.

Pertinent data obtained from expansion measurements on mortar bars containing aggregate composed of pure minerals and other selected materials are shown in Table II. In the case of igneous rocks, it has been necessary to ascertain what constituents comprise the reactive ingredients. It will be noted in Table II that three types of volcanic glass have expanded, obsidian and two pitchstones, the obsidian expanding the least in amount and the least rapidly. Bars containing Pyrex glass as aggregate have also expanded. On the other hand, volcanic glasses of basic composition and one glassy rhyolitic tuff have so far failed to expand. Several varieties of feldspar, the common varieties of ferro-magnesian minerals, and several minerals commonly occurring as accessories in igneous rocks (including two of the more common zeolites) have also failed to expand. Thus, in the case of the reactive acid to intermediate igneous rocks, suspicion has been directed away from the common rock-forming minerals and toward the glassy constituents.

TABLE II.—EXPANSIONS SECURED WITH SELECTED MATERIALS IN 1:2 MORTARS.

1 by 1 by 10-in. bars cured at 100 F. in sealed storage (Bureau of Reclamation Laboratory Tests)

Mineral	Age, months	Expansion, per cent		Percentage of Total Aggregate[a]
		1.42 per cent Alkali in Cement	0.18 per cent Alkali in Cement	
Limestone gravel (Colorado River)	4	0.080	−0.005	100
Siliceous limestone and chert gravels (Colorado River)	4	0.141	−0.011	50
Acid Volcanic gravels (Colorado River)	4	0.318	−0.003	50
Chert	7	0.121	−0.007	20
Obsidian	9	0.165	0.001	50 and 75
Chalcedony	8	0.254	−0.006	20
Friant andesite (includes meta-andesites)	10	0.339	−0.004	100
Friant andesite	5	0.353	−0.003	20
Brown pitchstone	9	0.446	0.005	50
Novaculite	8	0.517	0.002	75
Cowlitz andesite	11	0.546	0	50
Green pitchstone	8	0.602	0.002	50
Kimball acid volcanics	9	0.664	0.021	20
Kimball quartz rocks	10	0.066	−0.001	100
Kimball limestone	9	0.676	−0.012	100
Kimball chalcedony and opal	10	0.848	−0.001	100
Pyrex glass	6	0.841	−0.001	50
Opaline palagonitic tuff	12	0.920	0	5
Opal	9	2.075	−0.007	5
Opalized shale	5	2.348	−0.002	4.75
Opaline agglomerate	12	1.354	−0.002	5

[a] Remainder of aggregate is crushed quartz.

Microscopic examination of specimens of affected concrete discloses aggregate pebbles of igneous types which seem to have reacted with the concrete but which do not appear to contain glass. In such cases it may be that glass is present but overlooked because of its small amount or fineness of grain. However, some of these types, while not at present glassy, do contain substances which have formed from the devitrification of former glassy constituents; it may be that such devitrification products are also reactive. Similarly, it may be that normally nonreactive minerals, such as feldspars, may, if weathered or otherwise altered, give rise to alteration products which are themselves reactive.

If the formation of siliceous gels is an essential element in the "alkali reaction," it would seem evident that the reactivity of any given substance would be related to the abundance and the availability of silica in its composition. In some substances, such as opal and certain glasses, silica is both abundant and available. In many silicate minerals, such as feldspar, silica is abundant but apparently unavailable. The availabilities of the abundant silica in different cherts, flints, novaculites, chalcedonies, and related types is not yet established and it may develop that wide variations exist in each of these types.

As various theories have been advanced for the cement-aggregate reaction, companion laboratory tests of a chemical nature have been proposed to establish quickly the susceptibility of the suspected materials to the reaction. On the premise that magnesium carbonate in the aggregate reacted with the sodium hydroxide in the cement to form magnesium hydroxide and hydrated sodium carbonate, with accompanying increase in the volume of solid matter, Mr. Stanton at first presented a test to determine the soluble magnesium carbonate in an aggregate. Considerable discussion of this theory may be found in the literature, and Mr. Stanton apparently now feels that opaline silica is more probably responsible for the action encountered by him than the magnesium carbonate. Similarly, Bailey Tremper initiated experiments to study the expansion of specimens containing added alkali and exposed to alternate conditions of low and high humidity, on the assumption that volume changes accompanying hydration and dehydration of sodium carbonate, whether its source be the cement or aggregate, might explain the expansion. The Bureau of Reclamation has also conducted tests to determine the amount of alkali released from aggregates, following the procedure of Rengade, who suggested that aggregates containing high proportions of alkalies should be avoided; however, these results have been inconclusive and have not agreed with the service records of the materials or other laboratory tests.

Mr. Stanton has reported that he has for some time studied the solubility of aggregates in NaOH solutions, using volumetric methods to determine the degree of reaction. Later, considering that the earlier treatment was too severe, he experimented with weaker caustic solutions, but still without conclusive results. A similar test has been conducted by the Bureau of Reclamation, using sized aggregate and a gravimetric determination of dissolved silica. As would be expected, opal responds readily to the test, but for many aggregate types the results showed no correlation with service records or laboratory volume-change experiments.

Two somewhat similar methods have been proposed for the visual study of the reaction between cement and aggregate. Mr. Tremper has described results obtained by placing grains of aggregate on the surface of set cement paste, covering with water (and a film of oil to prevent carbonation), then allowing reaction to proceed. From the weight of gel formed, a measure of the reaction could be obtained. Mr. Stanton cast aggregate into a pat of neat cement, cured the specimen overnight, exposed the aggregate by lapping, and then immersed the pat in caustic solution. The extent of reaction was measured by observing the amount of gel formed around the aggregate grains.

Recently the Bureau of Reclamation laboratories have been pursuing a few additional lines of investigation which, although they have not resulted in anything conclusive, may be of interest. In an effort to detect the susceptibilty of various common rocks and minerals to attack by alkaline solutions, tests were initiated in which selected specimens were immersed in solutions of sodium carbonate, sodium sulfate, sodium hydroxide, and solutions leached from high-alkali cement, and cast within blocks of high-alkali, neat cement. Preliminary results suggest that solutions of sodium carbonate and of sodium sulfate have little effect during a seven-month period upon even those rock types highly reactive with high-alkali cements.

On the other hand, 1 *N* and 5 *N* solutions of sodium hydroxide have caused weight losses up to 23 per cent in certain acidic, volcanic rocks from the Bill Williams River (Parker Dam) aggregate. Significant attack on intermediate and basic volcanic rocks from the Bill Williams aggregate, and on pitch-stone, chalcedony, opal, and andesite from the Cowlitz River, Washington, has been observed. The losses in weight suffered by these rock and mineral types in the sodium hydroxide solutions have come about through differential chemical attack, alteration, and solution within the mass of the specimens, resulting in moderate to high porosities especially adjacent to the surfaces. At the completion of the tests all specimens will be carefully studied and all solutions and precipitates chemically analyzed.

It seemed possible that the susceptibilities to reactions might be measurable if finely ground powders of reactive materials were mixed as slurries with alkaline or acidic solutions. By way of experimentation along these lines, powders have been made (over 90 per cent through No. 200 sieve) of more than 40 rocks and minerals selected so as to be representative of all common rock and mineral types. These powders were respectively mixed in various proportions in dilute solutions of sodium hydroxide, hydrochloric, sulfuric, and acetic acids, and high- and low-alkali cement slurries, and the pH's of the resulting slurries measured immediately, and with passing time, by means of a Beckman pH meter. Changes in pH of the powder slurries due to additions of given amounts of strong acids or strong bases seem to be subtly related to the reactivity of the material in the powder, relatively smaller changes occurring with the more reactive substances. Many mechanical difficulties have been encountered in these procedures and in several instances ambiguous and anomalous results have been obtained, but it is felt that the results to date are sufficiently encouraging to justify continued research along these lines.

Additional microscopic studies have been inaugurated but have not yet progressed sufficiently to justify a report of results. These studies involve preparation of wet slides for the observation of cement hydration and of the effect of various cement slurries on finely powdered reactive aggregate materials. Polished surface studies of cement and concrete have been started with the objective of adapting the techniques, as developed by others, to the alkali problem.

Engineers are perennially confronted by the necessity of choosing aggregates and cements for current projects, and in spite of all the development of data which has resulted from field and laboratory studies, concrete engineers are nevertheless still inadequately armed for the task of making sound judgments with respect to these selections. The difficulties involved in making these practical day-to-day decisions resolve themselves into a few general categories. First, there is lacking a quick and positive test for potential reactivity of any given aggregate, and no rapid and certain method for evaluating in advance of use the relative compatibilities of specific cement-aggregate combinations. Second, the list of reactive aggregate materials embraces so many rock and mineral types that it is impracticable to reject materials which resemble others with known reactive histories. Third, very small amounts of known reactive substances appear in a large number of reactive aggregates, but we do not now know what is a tolerable limit of such substances below which amount they can be ignored and above which they must be reckoned with. Fourth, satisfactory uses of ameliorative admixtures or other preventive procedures, which might be applied in cases of doubt, have not been developed, although a promising approach may be through the use of pozzuolana cements. In short, from the standpoint of practical working procedures, we are not far advanced beyond our position of 1940, when the Bureau of Reclamation, the California Division of Highways, and a few other agencies took refuge in the specification of comparatively low-alkali cements for all major construction. Fortunately, the efficacy of thus specifying a limit for the alkali content has been proved.

Although it is clear that still lower alkali contents than heretofore specified would accomplish an additional improvement, and although laboratory experiments have indicated that, even with relatively low-alkali cements, certain fortuitous combinations can produce expansions, it is nevertheless true that in respect to structures in service, the reduction of the alkali content of cements has resulted in definite retardation, quantitative reduction, or complete elimination of the typical expansive deteriorations. In all instances in which the same reactive aggregates have been used with both high- and low-alkali cements, the structures or parts of structures in which low-alkali cements were used exhibited no expansion, or at least much less expansion, than where high-alkali cements were used. Only time will tell whether, in specific cases, the results of using low-alkali cement have been retardation of rate, reduction in amount, or complete elimination of the expansive reactions. But, in any case, the policy of specifying an upper limit on alkalies has been fully justified by service experience and laboratory tests, and that policy remains today the only practical working procedure of general applicability.

CONTRIBUTED BY W. C. HANNA[3]

Probably it is almost forty years since we first noticed "pop outs" in concrete which indicated that bad aggregates were being used. At first it was thought that possibly the "pop outs" were caused by some expansive extraneous material having been inadvertently mixed with the top coats in such concrete work as floors and pavements. Further experience and examinations showed us that with fresh "pop outs" we were usually able to find a small fragment of reactive shale at the bottom of the crater.

These "pop outs" would measure on the surface from as little as $\frac{1}{4}$ in. in diameter to as much as 3 in. in diameter, depending upon the size of the piece of reactive shale and the depth to which it was imbedded in the concrete. It was noticed that these "pop outs" were common where certain aggregates were used, and they apparently occurred in concretes made from any brands of cement, including foreign brands. Thus the penalty of using such aggregates was the liability of having "pop outs" in the concrete. It was interesting to notice that in most flat work the actual "popping" did not seem to continue after a few years, and that the concrete work was usually serviceable and satisfactory except for the unsightly "pop outs." Structures we have examined, as a rule, have not had any more cracks than is common in the eastern United States. The exception to these general conditions would be some work made with reactive aggregates and often bathed in sea water. This concrete was so "popped out," cracked, and disintegrated that the concrete was just about worthless. It was of particular interest in one of the last mentioned cases to note that the bad work was an extension to a good concrete wall made with good aggregates many years prior to the bad work.

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[3] Chief Chemist, California Portland Cement Co., Colton, Calif.

As early as 1934 we examined a structure which showed severe cracking and found gel formation and reactive aggregate. Continuing the investigation, we observed other structures in which such reactive aggregates had been used and which showed cracking without apparent regard for the brand of cement used. We followed the aggregates to their source and secured particles which reacted with saturated lime water and which produced a volume of gel estimated to be six to eight times that of the original particle.

We endeavored to make our experiences clear in our discussion of Thomas E. Stanton's paper (**7**)[4]. We will not repeat them here except to emphasize that any aggregate which will react in saturated lime water will certainly prove to be reactive in concrete, regardless of the alkali content of the cement used. We are of the opinion that the calcium hydroxide freed by water from any cement in any concrete must have an effect on the concrete and that the amount of other compounds of sodium, potassium, lithium, etc., will also have an effect.

The reaction between the aggregates and the cement may have a beneficial effect, although we are chiefly concerned with the action when disruptive forces are set up. Take for example the ordinary standard mortar briquets which show tensile strength of 700 to 1000 psi. at periods of 10 to 20 yr. when stored in water for 28 days and then in normal air. These specimens have such adherence between the cement and the Ottawa sand that the sand grains are broken in two at the time of testing. Our theory of the occurrence of beneficial reaction has not received general approval although no other reason for the high strength has been advanced.

We have found that some of the most reactive bad aggregates cease to be bad when they are pulverized, although they are then even more reactive. Our theory has been that there is beneficial reaction between the fine reactive aggregate particles and the compounds of calcium, sodium, potassium, and lithium. Indeed, it seems that it is necessary to have such compounds present in order to have good pozzuolanic action. This difference in reactivity between bad aggregates of different particle sizes may explain why some work may be good and other work poor. The large pieces of reactive aggregates cause the "pop outs" when they are near enough to the surface to do so; the smaller size of this aggregate seems to cause the cracks in concrete; and the very fine reactive dust will actually be beneficial when present with the other sizes.

The curing and exposure of concrete has a very decided effect upon the reactions. One concrete may be entirely satisfactory while another, which is the same except for the curing cycles, may be in decided distress. This same condition also applies to laboratory specimens, and extreme care must be used to have everything just the same where direct comparisons are to be made.

We have carried out an investigation of the expansion of mortar bars made with a reactive aggregate and a large number of cements of widely varying alkali content. These include the 132 commercial cements of the Medicine Lake sulfate resistance series which were furnished by Dalton G. Miller of the U. S. Soil Conservation Service. Chemical and physical data on 106 of these cements have been reported by Miller and Manson (5). Cements representative of all types and compositions are included in this series. The bars were made and stored under the conditions specified by T. E. Stanton.

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[4] The boldface numbers in parentheses refer to the bibliography appended to these notes, see p. 219.

At the age of 2 months the expansions of these bars, with few exceptions, were observed to be definitely related to the alkali content of the cements. At 6 months many exceptions were observed in this relation, and at the age of 18 months nearly all of the bars had expanded excessively. In many instances bars of low alkali content expanded to a greater extent than those of higher alkali content. No correlation was observed between the expansions and Miller's sulfate resisting rating. Some bars to which a deliberate addition of sodium hydroxide was made showed lower expansions than companion bars made without addition.

It may be thought by some that the excessive expansions at later ages occurred only as a result of particular proportions or sizings of the reactive aggregates. This explanation, even if correct, does not invalidate the data. There is reason to believe, however, that similar proportions and sizings of bad aggregate, may sometimes occur in actual practice. This being the case, excessive expansions may result to a degree quite comparable to that occurring in the test specimens. These data indicate that although a reactive aggregate and a low alkali cement may not show appreciable expansion in a few weeks, or even months, a dangerous possibility exists of ultimate excessive expansion regardless of the alkali content of the cement. Until such time as concrete structures made with known reactive aggregates and cements of known alkali content have attained a reasonable age, and a greater degree of correlation is shown between laboratory tests and ultimate service records, it is thought unwise to place undue reliance on the efficacy of limiting the alkali content of the cement as a means of protecting concretes made from reactive aggregates.

In making field examinations it must not be overlooked that alkalies may be present from sources other than the cement. It is well known that salt spray from the ocean is carried inland some distance. The total amount of alkalies in the aggregates and water used in concrete may greatly exceed the alkali content from the cement.

As a preventative for excessive expansion we have suggested the use of fine pulverized bad aggregate along with the cement where bad aggregate is suspected. We have also suggested the use of an addition of aluminum powder.

These statements should not be construed as indicating we are in favor of high-alkali cements. Any competent cement manufacturer knows that if his total alkali gets too high he may expect a variety of troubles. Our experience indicates that we may question the actual quality of any cement when we see the total alkalies migrate to a value of over 1 per cent. Comparisons have been made between cements with as high as 1.4 per cent alkalies and as low as 0.2 per cent alkalies. Our opinion is that cement with such a high alkali content cannot be classed as a normal cement.

We are particularly appreciative of the extensive program of work being directed by our chairman, R. F. Blanks. We know the pioneering work initiated by T. E. Stanton is going ahead in a logical manner. The work of Bailey Tremper and C. E. Wuerpel has been of great help in our attempts to get a balanced viewpoint of this problem. All investigators are to be commended for their willingness to advance theories on data which they know is meager. It is a pleasure to work with men who are not only willing, but even anxious, to listen to theories which may not in every way agree with their own.

CONTRIBUTED BY BAILEY TREMPER[5]

Investigations have been made in the laboratory of the Washington Department of Highways of the effect of variations in technique in making and storing mortar bars which have been commonly used for determining expansions of cement-aggregate combinations. Seemingly unimportant details of manipulation have been found to cause major differences in the amount of expansion. Until the method is standardized rigidly, agreement between different laboratories is likely to be nominal only. In fact, with highly reactive aggregates, large differences in expansion are not unusual among four bars fabricated from the same batch of mortar. By proper choice of details the relative degree of reactivity of two cements may be reversed although the same aggregates and storage temperatures have been used. Storage at 100 F. generally gives earlier and consistently greater expansion than storage at 70 F. One high-alkali cement has been found, however, which with a highly reactive aggregate, consistently gives much greater expansion under 70 F. storage than under 100 F. storage.

The mortar test bar is considered by the author to be a rough measure of the potential reactivity of cement-aggregate combinations. In his opinion, it cannot be used to predict precisely what will occur in job concrete employing the same cement and aggregate for the following reasons: the usual laboratory mortar mixture contains something like twice the quantity of cement that is used in average concrete and the concentration of alkalies per unit volume is doubled; only the fine aggregate, or the coarse aggregate crushed to fine sizes, can be used in the mortar bar and the increased surface area increases the rate and probably the extent of chemical reactions leading to expansion; moist storage conditions in the laboratory make for continued chemical reactions whereas in the field the concrete may be damp at intermittent periods only and then may be subject to loss of alkalies through efflorescence and leaching; on the other hand, the maintenance of uniform temperatures in the laboratory test may lead to relatively less expansion than will be experienced in the field, particularly if exposure is to freezing temperatures.

In spite of disadvantages in elapsed time and the uncertainty of interpretation, the mortar test bar remains the best method, so far developed, for determining the reactivity of cement-aggregate combinations.

In a previous paper (15), the author stated that further expansion in abnormal pavements and structures apparently had ceased at the end of about ten years. Subsequent observations, however, make it clear that expansion, or at least the visible effects of it, has not ceased. Relieving pressures at expansion joints in pavements has proved only partially successful as new offsetting and spalling due to end pressure in slabs have occurred at other joints. Portions of Clear Fork Creek Bridge that apparently were in good condition in 1940 (age of 10 yr.) have since developed the characteristic pattern of open cracking. Slightly younger structures in the same vicinity but at higher elevations have developed expansion and open cracking to a degree more serious than in the Clear Fork Creek Bridge. Structures constructed with cements presumably low in alkalies, however, continue to be free from visible defects.

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[5] Materials Engineer, State of Washington Department of Highways, Olympia, Wash.

Field observations and laboratory experiments make it appear that mildly reactive aggregates such as those from the Cowlitz River could be used successfully in less severe climates with cements of at least moderately high-alkali content.

The effect of severity of exposure, as measured by elevation above sea level, on durability of concrete has been observed in two series of structures in addition to the Cowlitz River group. While it is not yet entirely clear to what extent alkali-aggregate reactions have contributed to deterioration in these other groups, the outward appearance and occasional extreme expansion in handrails strongly suggest that trouble is due in part to this cause. Freezing and thawing is also an apparent cause. The degree of deterioration is directly connected with the severity of exposure as measured by elevation above sea level. Experiments are now under way in the laboratory to determine the effect on resistance to freezing and thawing of concrete containing suspected aggregates and cements of varying alkali contents. It is not anticipated, however, that answers will be obtained quickly because of the apparent necessity of allowing sufficient time for chemical reactions to progress within the concrete before subjecting it to cycles of freezing and thawing.

The problem of isolating the effects of possible alkali-aggregate reaction from simple lack of resistance to freezing and thawing, by means of field observations in Washington, is complicated by a number of factors. It is frequently difficult to determine the basic cause of a defect from observation of outward appearance. There is a lack of sufficient combinations of cements and aggregates and a disparity in ages in existing structures. There is insufficient knowledge of precipitation and temperature fluctuations which may vary greatly within short distances in mountain passes.

Finishing operations on concrete surfaces seem to influence greatly the early appearance of open surface cracking. Specimens to which a rubbed mortar finish have been applied have developed open surface cracking whereas similar specimens not so finished have not.

Regardless of the exact cause of the deterioration, observation of existing concrete structures in Washington makes the conclusion inescapable that over 90 per cent of the cases of unsatisfactory durability are those in which the cement was known or strongly suspected to have been high in alkali.

CONTRIBUTED BY CHARLES E. WUERPEL[6]

The interest and concern aroused in the concrete profession by the cracking of certain important structures in the United States, which has been attributed to a reaction between certain siliceous aggregates and the alkalies (Na_2O and K_2O) present in cement, resulted in a study of the phenomenon by this laboratory in connection with the Cement Durability Program authorized by the Office of the Chief of Engineers, U. S. Army. Membership on the Special Subcommittee on Effect of Alkalies in Cement on the Durability of Concrete, in the Society's Committee

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[6] Engineer-in-Charge, Central Concrete Laboratory, U. S. Army Corps of Engineers, Mt. Vernon, N. Y.

C-1 on Cement, has permitted the laboratory to keep in close touch with the results of studies made by other agencies, notably the Bureau of Reclamation, the California State Highway Department and the Washington State Highway Department. The relationship with the special subcommittee has included the exchange of specimens, reports, written discussions and photomicrographs. The specific investigations conducted by this laboratory have been based principally upon microscope inspection and chemical analysis of specimens of concrete obtained from sound and unsound structures in the East, upon suspected aggregates and upon the specimens included directly in the Cement Durability Program. Methods of observing the results of possible reaction have included those suggested by others, notably the measurement of the expansion of thin mortar bars, immersion of aggregates in sodium hydroxide solution and use of ultraviolet light.

The association of the author with this problem has led to the opinion that the problem of concrete failure by "alkali reaction" may be one of colloid chemistry. The hypothesis which postulates that disruptive stresses are developed solely by a product of reaction between certain types of aggregate and the alkali content of some cements becomes less plausible as the results of various investigations are reported. This opinion was formed in view of (1) the apparently conflicting results that are being obtained in some investigations of high-alkali cements and reactive aggregates, (2) the absence of evidence of any reaction products other than the soft gelatinous exudations observed on the surfaces of certain unsound concretes, and (3), perhaps, the inability of the author to observe any physical manifestation of reaction which could be considered to cause material expansion in concrete.

There can be no doubt that certain aggregate types react to a variable degree with a cement hydrate, and that other aggregate types are metamorphosed in the environment of a cement hydrate, but no evidence has been observed that would indicate that such reactions were the direct cause of concrete failure. To the contrary, some reactions of this nature might be beneficial in strengthening the bond between the aggregate particle and its surrounding medium. Opaline silica and, perhaps to a lesser degree, certain volcanic glasses appear to be the exceptions. The detrimental effects of their presence cannot now be questioned. The general absence of opal in the aggregate in the northeastern section of the country has not permitted extensive study here of exudations or of opaline silica and their influence on unsoundness in the specimens that have been collected from concrete structures in the northeastern United States. Therefore, the opinions expressed are influenced to a considerable degree by the reported observations of others coupled with less extensive studies made at this laboratory on a few specimens received from the West.

It has been shown by Stanton and by other investigators that opaline silica and certain volcanic glasses are readily affected by solutions of sodium hydroxide. It has also been shown that mortar bars containing certain percentages of opaline silica or volcanic materials expand excessively at early ages with cements of relatively high alkali content. On the other hand, it has been reported recently by Hanna that mortar bars made with opaline silica and cements of comparatively low alkali content are expanding to a greater ultimate degree, but at later ages, than bars with a higher alkali cement. Such results indicate that the predominant factor is the opaline silica itself and not necessarily the alkali content of the cement. When the alkali content is optimal, reaction takes place in a relatively short period of time. When the alkali content is lower, the reaction still takes place, but at a slower rate and requires a longer time to become evident. It is considered possible that the caustic action of the calcium in cement may itself be sufficient in time to cause a reaction with opaline silica contained in aggregate.

Visible evidence of reaction appears to be limited to the development of gelatinous exudations within or on the surfaces of the unsound concrete. No other evidence of a reaction product has been reported. Chemical analyses of these exudations show them to be variable in composition, but to be always very high in silica. This indicates that they are not necessarily sodium silicates at all but are more likely silica sols which have adsorbed variable quantities of the ions of sodium, potassium, calcium and other elements. The sols coalesce, become gelatinous matter or gels on the release of pressure or on coming in contact with the air. The fact that these exudations appear in greatest amounts on concrete in which opaline silica is present is a strong indication that they are derived largely from this material.

Opal is a natural silica gel that has hardened over a long period of time and may be returned to the gelatinous condition in the presence of certain alkali solutions. It is not unreasonable to expect that it will break down in alkaline cement waters to form a gelatinous substance. A similar substance has been observed in a more or less liquid state within concrete structures of appreciable age. On one occasion a drop of gelatinous liquid, which hardened rapidly in contact with the air, was observed in a specimen taken from a sound concrete structure twelve years old. This appears to indicate that the gelatinous substance remains soft and fluid indefinitely so long as it is confined under pressure within the concrete and away from the air. It seems that this substance can be eliminated as the direct source of a disrupting force sufficiently great to cause concrete failure.

The gain in strength of hydrating portland cement is taken to be due largely to the hardening of the hydrous calcium silicate gels formed by the reaction between water and the compounds in the cement. The hypothesis is presented that the hardening process of the cement gels may be disrupted and thrown out of balance when invaded by foreign sols from the aggregate. Localized acceleration or retardation of coalescence may develop, causing internal stresses which slowly result in concrete failure as the amorphous substances become less and less elastic. Or, the molecular balance of the hydrous calcium silicate may be upset causing additional molecules of water to combine with the calcium silicate with a residual expansion in volume of the hydrated or hydrating product.

Colloidal silica, such as that derived from the disintegration of opal, readily adsorbs any available sodium ions, the presence of which greatly reduces the tendency of the colloidal particles to coalesce. At first, this condition will be localized close to a particle of opaline silica. Limited amounts of the silica from the opal may combine with siliceous hydrates of the cement, but other portions may slowly migrate through the groundmass of the concrete, adsorbing other available ions and influencing the coagulation of the areas of cement hydrate through which they pass. As a colloidal solution, silica "will diffuse through animal, collodion, or parchment paper membrane, sufficiently fine in texture to retain colloidal silver...... under reduced pressure, it becomes unstable and finally undergoes a change in which viscosity rises rapidly and the entire mass sets to a solid gel" (26). Just how the influence of this variable in impure silica sols may affect the normal hydrous calcium silicate gel of the cement is a problem in colloid chemistry, and it is believed that a study of this field must be made before a complete explanation of the expanding and cracking phenomena observed in certain western structures can be made and the effects remedied.

There remains much to be learned of the chemistry and physics of portland cement and of the minerals in aggregate which may react with the hydrated product of portland cement. It may be that the policy of specifying a nominal upper limit on alkalies is justified when aggregate known to possess opaline silica must be used, but the indications that even lower quantities of alkali in cement may, in combination with certain aggregates, cause very considerable expansion in concrete at later ages makes such a policy a tenuous stop-gap. If the alkali content of portland cement must be reduced, means for such reduction must be perfected by the manufacturers in such a way that the other properties of the cement will not be affected adversely. A prematurely adopted limitation on the alkalies might cause an undesirable influence in the manufacture of portland cement.

In summation, it must be concluded that the existence of the expansive cracking phenomenon in a number of structures in the West presents a challenge to the chemists, physicists, and geologists to improve their knowledge of the chemistry and physics of concrete. The challenge has been recognized but the victory is not made complete by a moderate limitation of the alkalies (Na_2O and K_2O) in portland cement.

CONTRIBUTED BY F. H. JACKSON[7]

In the study of the reaction between high-alkali cements and certain types of concrete aggregates, the Public Roads Administration has been primarily interested in two phases of the problem: first, the identification of aggregates which have given poor results in concrete under conditions indicating the possibility of chemical reaction; and, second, the study of accelerated methods of identifying alkali-reactive aggregates. Samples of cement and aggregates from many locations where chemical reaction is suspected have been obtained for study, and specimens prepared for both expansion determinations of mortar in moist air storage and accelerated tests of mortar and aggregates.

Preliminary volume change tests of 1:2 mortars were made using the siliceous magnesium limestone identified by Stanton (7) as a highly reactive material. The behavior of mortar containing this aggregate followed closely the results obtained by Stanton (7). Small amounts of the limestone in inert sand furnished an aggregate which gave excessive expansion with high-alkali cement and negligible expansion with low-alkali cements. This would seem to indicate that the alkali content of the cement is definitely a factor in the reaction. Increase in the limestone content above 10 per cent usually resulted in a great reduction in the amount of expansion. In one case, however, a sand containing 25 per cent of limestone passing the No. 30 sieve gave greater expansion at ages of 28 to 52 weeks than any other combination tested. This is unusual as most of our tests have indicated that the use of as much as 25 per cent of the reactive limestone actually reduces expansion.

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[7] Principal Engineer of Tests, Public Roads Administration, Federal Works Agency, Washington, D. C.

An intensive study of aggregates from the northwestern states was made to identify sources of aggregates showing reaction and, in the case of gravels, what components of the material are reactive. A number of sources of aggregates have been found to contain definitely reactive materials. Exact identification of the reactive materials has not yet been completed, but it appears that the acidic extrusive igneous rock types are more likely to react with high-alkali cements than basic extrusive or intrusive types.

It has been stated that the excessive expansion observed in some concrete pavement made with sand-gravel from the Platte River valley in Nebraska may be due to the presence of small percentages of opal in the aggregate. It has also been stated that the feldspar constituent of this aggregate might be reactive. Some preliminary laboratory work has been done on this problem in addition to the tests reported in the paper presented during the 1942 meeting of the Highway Research Board (20). A series of mortar specimens was prepared with high-alkali cement and aggregates consisting of the normal Platte River sand-gravel, gravel composed entirely of feldspar, and sand-gravel containing various percentages of opal. The results of these tests show that to an age of two months only those mortars containing opal have developed excessive expansion. It appears that the opal content of the Platte River sand-gravel may be responsible for at least some of the expansion found in this concrete.

In the southeastern states, some trouble with concrete containing blast-furnace slag in combination with high-alkali cements has been reported. To determine if blast-furnace slag from this and other areas is subject to alkali reaction, mortar tests were made using a number of different slags crushed to sand size. In no case was any unusual expansion obtained to an age of one year, and it is believed that blast-furnace slag, of the grade prepared for use in concrete, is not reactive with high-alkali cement.

Expansion tests were made of mortars prepared with high-alkali cements and representative samples of the quartz or limestone and sandstone sands used extensively in New York and Pennsylvania. To an age of one year, little expansion attributable to chemical reaction has been found.

Attempts were made to accelerate the expansion of high-alkali cement mortars by storage of the test specimens in a 1 per cent solution of sodium hydroxide at 70 F. Specimens stored under these conditions expanded almost twice as much as duplicate specimens stored in moist air at a temperature of 130 F. However, some unusual results were found in the case of certain materials. Duplicate specimens were prepared with high- and low-alkali cements and an inert sand containing 10 per cent siliceous magnesium limestone. These were placed in the solution at an age of 48 hr. To an age of 12 weeks, both sets of specimens behaved normally, the high-alkali cement specimens expanding at a fairly constant rate and the low-alkali cement specimens showing only a trace of expansion. At some period between 12 and 17 weeks, the specimens made with low-alkali cement started expanding at a rapid rate. At an age of 38 weeks the low-alkali cement specimens showed the same percentage of expansion as those made with high-alkali cement, and at an age of one year the former had almost one and one-half times as much expansion as the latter. The behavior of this low-alkali mortar is so different from that found for similar mortar in moist air storage that it seems apparent that the alkali in solution entered the reaction. Further study of specimens subjected to this type of storage is in progress.

In his report of December, 1940, Stanton (7) suggests that a rapid test for alkali reactive aggregates could be made by immersing the aggregate in a 10 per cent solution of sodium hydroxide. The appearance of a fluffy white exudation on the surface of the aggregate within a few days would be presumed to indicate that the material would probably be reactive with high-alkali cement. This test was made on a group of almost 600 aggregates, over 400 of which are from the eleven western states. Of this group, only 59 give evidence of being reactive. These range from shale and pumice to granite and basalt. Mortar expansion tests with high-alkali cement on these samples show only one to develop excessive expansion.

Study of a pat test proposed by Stanton (25) is now in progress. In this test the aggregate is placed in a pat of meat cement and after the latter has hardened the aggregate is exposed by grinding the face of the pat. The pat is then placed in a 0.5 *N* solution of each of sodium hydroxide and potassium hydroxide in saturated lime water. Alkali reaction is noted by the appearance of an exudation on the exposed face of the aggregate. To date, little correlation has been obtained between the results of this test and the results of a test for expansion of mortar. In a number of cases, exudations have been found on pats prepared with the same aggregate and both high- and low-alkali cements, indicating that the reaction may be between the aggregate and the alkali in solution rather than the alkali in the cement. In spite of this, it is believed that the method shows some promise as an indicatory test, but it appears desirable to reduce the strength of the solution. A solution containing 0.5 per cent of each of the hydroxides is suggested.

Some study of the method proposed by Tremper (15) involving the use of ultraviolet light to obtain fluorescence has been made. The results have not been entirely satisfactory. Some of the materials which show a definite reaction with high-alkali cement fail to fluoresce, and a number of aggregates which do not react with the cement do react with the ultraviolet light.

Several series of tests have been made to devise means of neutralizing the alkali in cement. Ground silica and diatomaceous earth have been found ineffective in amounts of 5 per cent by weight of the cement or less. The use of 2 per cent of calcium chloride by weight of the cement likewise failed to diminish the expansion but 4 per cent of sodium sulfate caused a great reduction in the expansion to an age of 36 weeks. Expansion then began and, to an age of 78 weeks, is continuing at about the same rate as is found in untreated mortar.

At the present time, there appears to be no accelerated method of test which can be relied upon to indicate definitely the materials which are reactive with high-alkali cement. The pat test is of some value, however, as any material which fails to react under the conditions of this test probably will not react with alkali in service. The test could thus be used as an acceptance test in routine work. For quantitative measurements, the storage of mortar bars in moist air appears to be the only satisfactory test now available. Continued study of the storage of mortar specimens in a 1 per cent solution of sodium hydroxide will be made in the hope that the method can be used with the storage period limited to 28 days.

CONTRIBUTED BY F. B. HORNIBROOK,[8] H. INSLEY,[8] AND L. SCHUMAN[8]

Studies at the National Bureau of Standards of the reactions of various aggregates with high-alkali cements may be summarized as follows: sands, gravels, and crushed stones from a number of different sources have been tested for expansivity, using cements of high soda, high potash, or low alkali content and various other combinations. In addition to those containing opal, only those materials containing volcanic glasses have shown abnormal expansion. In many cases, the expansion is very slow in 70 F. storage but much faster at 100 F., or with alkali added to a high-alkali cement. Expansions comparable to those obtained with high-alkali cements have been found with low-alkali cement to which chlorides or sulfates of potassium or sodium were added. It was also found that addition of these salts to an aqueous extract of cement raised the pH of the solution the same amount as the addition of an equivalent amount of the hydroxides of sodium or potassium. Ground firebrick, composed principally of the form of silica known as tridymite, gives expansion comparable to that obtained with opal, especially in 100 F. storage. A number of minerals and rocks including feldspars, chalcedony, hornblende, rhyolite, etc., have also been tested, as well as the natural glasses obsidian and pitchstone. Only the latter shows significant expansion with a high-alkali cement. In practically all tests, K_2O causes much more rapid expansion than Na_2O.

Considerable work has been done on etching tests with alkalies of polished surfaces of aggregate material. The reactivity is determined by examination with the reflecting microscope. Much work remains to be done. It appears, however, that materials which show a microscopically visible etch after 17 hr. in a 10 per cent NaOH solution at 50 C. will also give evidence of considerable expansion when combined with a high-alkali cement and tested for expansion of mortar bars. Opaline aggregates will show a very marked etch, many aggregates containing volcanic glass will show a noticeable but less marked etch. Chalcedony does not etch under these conditions but shows a marked etch at higher temperatures (90 C.). Cherts composed of microcrystalline quartz do not etch even at 90 C. for long periods of time.

Various minerals stored for one year in 5 per cent solutions of NaOH and KOH respectively resulted in approximately the same ranking of degree of reaction as was obtained by autoclaving the minerals in the same strength solution for one to four hours. The only minerals that were appreciably affected were opal, chalcedony, and pitchstone, listed in descending order of degree of reaction.

While current practices in testing of combinations of cements and aggregates may probably be adequate for determining the potential reactivity of a given aggregate with a high-alkali cement, further work is needed to ascertain the limits of concentration of alkali and reactive mineral above which concentration disintegration may be expected to occur under given exposure conditions. In addition, if the mechanism of disintegration could be explained in detail, real progress could probably be made in adopting steps to render compatible the only aggregates economically available in certain areas with cements of medium alkali content. It appears, accordingly, that considerable effort should be concentrated on shedding light on the full details of the mechanics of disintegration.

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EDITORIAL NOTE

The subject of "Effect of Alkalies in Portland Cement on the Durability of Concrete" is further discussed in a paper by T. E. Stanton, "Studies to Develop an Accelerated Test Procedure for the Detection of Adversely Reactive Cement-Aggregate Combinations," see p. 875, together with discussions thereof by W. C. Hanna and F. B. Hornibrook.

REPORT OF COMMITTEE C-5

ON

FIRE TESTS OF MATERIALS AND CONSTRUCTION

Since the latest report of Committee C-5 on Fire Tests of Materials and Construction in 1941, one meeting was held on April 9, 1943, in New York, N. Y.

The committee has recently lost one member by resignation: United Engineers and Constructors, consumer, represented by W. E. Belcher. This resignation was accepted at the meeting on April 9, 1943.

The loss by death of M. L. Carr, representative of Pittsburgh Testing Laboratory, and E. F. Hartman of Protexol Corp., two active, interested members, is a matter of sincere regret.

A paper on "Tests of the Fire Resistance and Thermal Properties of Solid Concrete Slabs and Their Significance," has been prepared under the auspices of Committee C-5 for presentation at this annual meeting.[1] The author is Carl A. Menzel, Associate Engineer in the Research Laboratory of the Portland Cement Assn., who describes a series of fire tests on solid concrete slabs varying in thickness and in the materials used in their construction. The tests were conducted in accordance with A.S.T.M. Standard Methods of Fire Tests of Building Construction and Materials (C 19 - 41) except as noted in the paper, for the size of specimen, which deviation may have essential effects in point of stability and integrity of the constructions as exposed to fire. The results are recorded to show the effect of variation in the size of aggregate, of duration of drying of slabs before testing, of relationship between thickness of slab and fire endurance period, of heat transmission and other features. The results are discussed and certain conclusions enumerated.

I. New Tentative Standard

On the recommendation of its Subcommittee III on Fire Tests of Wall Opening Protectives (A. L. Brown, chairman), Committee C-5, as a result of action taken at the meeting on April 9, 1943, recommends for publication as tentative new Methods of Fire Tests of Window Assemblies.[2] These methods are the result of the continuance by the subcommittee of its study of fire tests for the protection of wall openings. They cover fire tests for window or door assemblies consisting of or containing elements of glass or other materials for the transmission of light.

The first of the series of tests of opening protectives to serve against the passage of fire, heat, fumes, or smoke was presented to the Society in 1940, and in 1941 was adopted as standard. These Standard Methods of Fire Tests of Door Assemblies (C 152 - 41) have since been reviewed by ASA Sectional Committee A2 on Specifications for Fire Tests of Materials and Construction and on its recommendation were approved as American Standard by the American Standards Association and assigned the ASA designation A2.2—1942.

The new proposed methods of fire tests of window assemblies have been developed to meet the needs arising in situations differing from those for which the earlier standard had been prepared.

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[1] See p. 1099.

[2] The recommendation for acceptance as tentative of these methods failed of approval at the annual meeting of the Society, see Summary of Proceedings, p. 7.

II. TENTATIVE STANDARD CONTINUED AS TENTATIVE

At the annual meeting of the Society in June, 1941, the Tentative Method of Test for Fire-Retardant Properties of Wood (C 160 – 41 T), prepared by Subcommittee II on Fire Tests of Lumber (W. J. Krefeld, chairman) was accepted by the Society, for publication as tentative. The subcommittee has reported that although no objections or criticisms have been received since the method was published a year or more ago, it seemed desirable to continue it in its present status. The method is now being applied to emergency work, and this experience in its application will be helpful in further consideration by the subcommittee. This conclusion by the subcommittee was unanimous.

On the recommendation of the subcommittee, confirmed by letter ballot of Committee C-5, it is recommended that Tentative Method C 160 – 41 T be continued as tentative for another year. The result of the letter ballot in Committee C-5 showed that of the 29 votes cast, 26 were for continuation as tentative for another year and 3 for adoption as standard.

III. WITHDRAWAL OF TENTATIVE STANDARD

The Tentative Specifications for Fire-Retardant Properties of Wood for Scaffolding and Shoring (C 132 – 40 T) were accepted for publication as tentative in 1940. Subcommittee VI on Fire Tests of Scaffolding (W. B. White, chairman), which had prepared these specifications, not having received any adverse comments or criticisms, recommended their adoption as standard. This recommendation, however, was rejected by Committee C-5 at its meeting on March 5, 1941, on the grounds that such action was not yet appropriate because "the small difference in weight loss in the timber test between well-treated and untreated material makes it necessary to set the limit at a figure that can be met by untreated wood," and because "other methods of testing might be adopted for purposes of these specifications." The specifications were accordingly continued as tentative for another year.

Early in February, 1943, Subcommittee VI recommended, by a vote of five out of seven, two members not voting, that the specifications be adopted as standard. But, inasmuch as the Tentative Method of Test for Fire-Retardant Properties of wood (C 160 – 41 T) developed in 1941 is suited for determining the fire-retarding properties of wood used for scaffolding and shoring, three questions were submitted by letter ballot to the members of Committee C-5 relative to Tentative Specifications C 132 – 40 T: namely, (*1*) Shall the specifications be recommended for adoption as standard? (*2*) Shall they be continued as tentative for another year? (*3*) Shall they be withdrawn and referred back to Subcommittee VI for further consideration?

From a membership of 32, 29 members returned their ballots, 3 of whom voted for adoption as standard; 6 for continuation as tentative; and 20 (the required two-thirds vote) for withdrawal. In view of this, at the meeting of Committee C-5 on April 9, 1943, it was unanimously voted to recommend to the Society that the Tentative Specifications for Fire-Retardant Properties of Wood for Scaffolding and Shoring (C 132 – 40 T) be withdrawn.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 32 members; with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Fire Tests for Window Assemblies[1]	24	0	4
II. TENTATIVE STANDARD CONTINUED AS TENTATIVE			
Test for Fire-Retardant Properties of Wood (C 160 - 41 T)[a]	26	3	1
III. WITHDRAWAL OF TENTATIVE STANDARD			
Test for Fire-Retardant Properties of Wood for Scaffolding and Shoring (C 132 - 40 T)[b]	20	9	1

[a] The classified vote on the continuation as tentative of the Tentative Method of Test for Fire-Retardant Properties of Wood (C 160 - 41 T) was as follows: Affirmative: 11 producers, 4 consumers, 11 general interests; negative: 1 producer, 2 consumers.

[b] The classified vote on the withdrawal of the Tentative Method of Test for Fire-Retardant Properties of Wood for Scaffolding and Shoring (C 132 - 40 T) was as follows: Affirmative: 7 producers, 4 consumers, 9 general interests; negative: 3 producers, 3 consumers, 3 general interests.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Fire Tests of Wall Opening Protectives (A. L. Brown, chairman) held a meeting on the morning of April 9, 1943, and discussed in detail the preliminary draft (March 26, 1943) of the proposed Tentative Methods of Fire Tests of Window Assemblies. At this meeting the subcommittee considered the revised scope of the proposed method and also several changes in the preliminary draft. The changes concern principally the sections dealing with the recording of temperatures on the unexposed side, size of test assemblies, application of the hose stream, dislodgment of glass lights, and conditions of acceptance. The subcommittee recommends that the revised Methods of Fire Tests of Window Assemblies be accepted for publication as tentative.[2]

Subcommittee IV on Fire Tests of Acoustical and Similar Finishes (S. H. Ingberg, chairman) held a meeting on April 9, 1943, at which consideration was given to the results of fire tests conducted according to the Federal Specification SS-A-118 for Prefabricated Acoustical Units (formerly SS-T-302) by Underwriters' Laboratories, Inc., Columbia University, and the National Bureau of Standards. A discussion of the several methods resulted in a decision by the subcommittee to restrict future consideration to the development of the tunnel type tests used by Underwriters' Laboratories, Inc., and the National Bureau of Standards. Tests of this type are designed to produce data on flame spread, fuel contributed, and smoke generated. The subcommittee has no definite recommendation to make at this time, pending the accumulation of further test data by the tunnel test method.

Subcommittee VII on Relationship of Sizes of Test Samples (S. H. Ingberg, chairman) reports that, in general, tests have demonstrated that the sizes of test samples at present specified are acceptable, but that in certain cases smaller samples had been used in preliminary or pilot tests, and that in others composite samples, embodying more than one construction, had been utilized in a single standard size test. Where data on temperature transmission only are desired, the necessary information could probably be obtained in either the smaller or the composite samples.

The subcommittee has no recommendation to make at this time.

This report has been submitted to letter ballot of the committee which consists of the 32 members; 28 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. P. MILLER,
Chairman.

H. M. ROBINSON,
Secretary.

REPORT OF COMMITTEE C-7

ON

LIME

Committee C-7 on Lime held one meeting during the year, on June 24, 1942, in Atlantic City, N. J. By letter ballot of the committee it was decided not to hold a meeting in conjunction with the Spring Group Meetings in Buffalo, N. Y. One new member has been added to the committee during the year, and two changes in representation have occurred and one additional representative has been appointed. One consumer member has been reclassified as a general interest member. The total membership at present is 56, 26 of whom are classified as producers, 6 as consumers, and 24 as general interest members.

Subsequent to the 1942 annual meeting, Committee C-7 presented to the Society through Committee E-10 on Standards a tentative revision of the Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (C 25 – 29). This tentative revision was accepted[1] by Committee E-10 on August 24, 1942, and appears in the 1942 Book of A.S.T.M. Standards, Part II, p. 1439. Committee C-7 recommends that this revision be continued as tentative during the coming year.

The subcommittees are actively engaged on a number of projects involving certain standards for which the committee is responsible, but no definite revisions are advocated at this time. The other specifications still are considered in accord with present practice and require no revision. Committee C-7 therefore recommends to the Society that all of the standards and tentative standards under its jurisdiction be continued in their present status without revision.

New specifications are being considered for newer types of hydrated lime for structural purposes and methods for testing them.

This report has been submitted to letter ballot of the committee which consists of 56 members; 34 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. C. Voss,
Chairman.

L. K. Herndon,
Secretary.

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee C-7 reported results of the letter ballot vote as follows: Of a total membership of 55, 31 members returned their ballots, of whom 21 voted affirmatively, 2 negatively, and 8 members marked their ballots "not voting."

REPORT OF COMMITTEE C-8

ON

REFRACTORIES

Committee C-8 on Refractories held one meeting during the year, at the Dearborn Inn near Detroit, Mich., on August 27, 1942, in conjunction with the meeting of the Refractories Division of the American Ceramic Society.

Three new members were elected to the committee during the year: C. B. Bradley, representing Johns-Manville Corp., to replace G. A. Downsbrough; John Lowe, representing the American Foundrymen's Assn., to replace J. R. Allan; and J. W. Whittemore as representative of the American Ceramic Society.

E. H. Van Schoick, chairman of the Subcommittee IX on Classifications, died in March, 1943. The Advisory Board has appointed S. M. Swain as the new chairman of Subcommittee IX.

The committee was active during the year. The Editorial Subcommittee prepared a new edition of the Manual of A.S.T.M. Standards on Refractory Materials for publication in 1943. In order to include as many standards as possible in the new Manual, the following recommendations, resulting from actions of the committee at its meeting on August 27, 1942, and subsequent letter ballot, were submitted to the Society through Committee E-10 on Standards, all of which were accepted.

I. New Tentative Standards Accepted by Committee E-10

Tentative Specifications for Air-Setting Refractory Mortars (Wet Type) for Boiler and Incinerator Services (**C 178 – 43 T**).[1]—There has been a need for some time for a specification for air-setting refractory mortars, and these are the first such specifications presented for publication by Committee C-8. They were accepted by Committee E-10 on March 20, 1943, and assigned the designation indicated.

Tentative Method of Test for Sieve Analysis and for Water Content of Wet Refractory Materials (**C 92 – 43 T**).[1]—The Standard Method of Test for Particle Size of Ground Refractory Materials (C 92 – 36) had been on the books of the Society for six years without revision. Committee C-8 made a number of suggested and desired changes, and recommended that Standard Method C 92 be withdrawn and replaced by a new tentative method. The essential changes in the method are: Outlining under "scope" the features and usefulness of the wet and dry methods of sieve analysis; incorporating in the method a procedure for determining the water content of refractory products in the wet condition; and making a careful, thorough revision of the description for carrying out the operation of the test methods. This recommendation was accepted by Committee E-10 on March 20, 1943. Standard Method C 92 was accordingly withdrawn and the new tentative method issued under the designation C 92 – 43 T.

Tentative Methods of Test for Combined

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[1] Manual of A.S.T.M. Standards on Refractory Materials, June, 1943; also 1943 Supplement to Book of A.S.T.M. Standards, Part II.

Drying and Firing Shrinkage of Fireclay Plastic Refractories (**C 179 - 43 T**),[1] *Panel Test for Resistance to Thermal and Structural Spalling of Fireclay Plastic Refractories* (**C 180 - 43 T**),[1] *and Workability Index of Fireclay Plastic Refractories* (**C 181 - 43 T**).[1]—The Tentative Specifications for Fireclay Plastic Refractories for Boiler Furnaces and Incinerator Service (C 176 - 42 T), accepted last year, included test procedures for obtaining the drying and firing shrinkage, the workability index, and for carrying out the panel spalling test on this type of refractory material. The committee felt that these methods should be separated from Tentative Specifications C 176, and accordingly recommended that the above three methods be accepted for publication as tentative. They were accepted by Committee E-10 on March 20, 1943, and assigned the designations indicated.

Tentative Method of Test for Thermal Conductivity of Insulating Fire Brick (**C 182 - 43 T**).[1]—For some time there has been a desire for a standard method which can be used in the refractories industry for obtaining reproducible results in thermal conductivity tests. The committee selected, after careful consideration, the Globar-heated calorimeter equipment as developed by C. L. Norton, and recommended it for publication as tentative for use as applied to insulating refractories only. The new tentative method was accepted by Committee E-10 on April 15, 1943, and assigned the designation indicated.

II. TENTATIVE REVISIONS OF STANDARDS ACCEPTED BY COMMITTEE E-10

Standard Method of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (**C 38 - 42**).[2]—In order to conform to present-day practice, the committee recommended certain revisions in Standard Method C 38 for publication as tentative. The revision requires the specimen to be weighed to the nearest 0.05 lb. rather than 0.1 lb. now specified, also the permissible variation in temperature is being revised to a value of ± 20 F. (± 10 C.) instead of "plus 35 F. and minus 45 F. (plus 20 C. and minus 25 C.)." The last three sentences in the section on dismantling test panel are being deleted, since they refer to a matter which is now obsolete. This revision was accepted as tentative by Committee E-10 on March 20, 1943.

Standard Method of Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (**C 107 - 42**),[2] *and Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick* (**C 122 - 42**).[2]—For several years the permissible range of furnace gas pressure during preheating has been 0.3 to 0.6 in. of water in Standard Methods C 107 and C 122. Experimental work has shown that a small variation in furnace gas pressure is responsible for an appreciable increase or decrease in spalling. It has also been found practical to maintain the furnace gas pressure to within narrower limits. It was recommended, therefore, in the interest of refining the test as much as possible, to revise Section 4 (*d*) of each of these two methods, to require that the furnace gas pressure during preheating "shall be within the limits of 0.4 to 0.5 in. of water," at the same time deleting the note which follows this section. Previously it was recommended that when oil was used as fuel, the gas furnace pressure should be maintained between 0.1 and 0.2 in. of water. It has been demonstrated that furnaces heated with oil can now operate within the recommended limits of gas furnace pressure.

These proposed revisions in Standard

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[2] 1942 Book of A.S.T.M. Standards, Part II.

Methods C 107 and C 122 were accepted by Committee E-10 on March 20, 1943, for publication as tentative.

Standard Method of Test for Pyrometric Cone Equivalent of Refractory Materials (**C 24 – 42**).[2]—The committee recommended changes in the section on preparation of test cones which include: subjecting the samples to a preparatory heat treatment in an oxidizing atmosphere to prevent bloating of the material when made into cones and subsequently heated to the softening point; and presentation of the dimensions and shape of the standard cone in the form of a drawing. Since it was found impracticable to make standard cones with the length of the side of the truncated tip as small as $\frac{1}{32}$ in., a new measurement of 0.075 in. was chosen because this represents the present practice. These revisions were accepted for publication as tentative by Committee E-10 on March 20, 1943.

Standard Definitions of Terms Relating to Refractories (**C 71 – 42**).[2]—Committee C-8 recommended that the following definition of the term "calcining" be accepted for publication as a tentative revision of Standard C 71 to replace the definition issued in June, 1935 and modified in June, 1942.

> *Calcining of Refractory Materials.*—The heat treatment to which raw refractory materials are subjected, preparatory to further processing or use, for the purpose of eliminating volatile chemically combined constituents and producing volume changes.

This revised definition was accepted as tentative by Commiteee E-10 on March 20, 1943.

These several recommendations have been submitted to letter ballot of the committee which consists of 38 members; 35 members returned their ballots, with the results shown in Table I.

All of these recommendations of Committee C-8 concerned tentative standards or tentative revisions and since they have been accepted by Committee E-10, the committee has no additional recommendations on standards to make to the Society at this time.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE ON RECOMMENDATIONS ACCEPTED BY COMMITTEE E-10.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Tentative Standards			
Spec. for Air-Setting Refractory Mortars (Wet Type) for Boiler and Incinerator Services (C 178 – 43 T)	31	2	2
Test for Sieve Analysis and for Water Content of Wet Refractory Materials (C 92 – 43 T)	34	0	1
Test for Combined Drying and Firing Shrinkage of Fireclay Plastic Refractories (C 179 – 43 T)	34	0	1
Panel Test for Resistance to Thermal and Structural Spalling of Fireclay Plastic Refractories (C 180 – 43 T)	33	0	2
Test for Workability Index of Fireclay Plastic Refractories (C 181 – 43 T)	33	0	2
Test for Thermal Conductivity of Insulating Refractories (C 182 – 43 T)	27	1	7
II. Tentative Revisions of Standards			
Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (C 38 – 42)	35	0	0
Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 – 42), and of Super Duty Fireclay Brick (C 122 – 42)	30	2	3
Test for Pyrometric Cone Equivalent of Refractory Materials (C 24 – 42)	35	0	0
Def. of Terms Relating to Refractories (C 71 – 42)	33	1	1

Activities of Subcommittees

Subcommittee I on Industrial Survey (L. C. Hewitt, chairman) has completed an Industrial Survey of Refractory Service Conditions in Electric Steel Furnaces. This survey, prepared by Edward E. Callinan, has been accepted and appears as an Appendix to this report. I. W. Moe, who was undertaking the preparation of the survey of refractories in the oil industry, was unable to finish this assignment because of other duties, but the work he compiled was turned over to the committee, and one of the associates of F. A. Harvey at the Harbison-Walker Refractories Co. will complete it. The survey on bottle glass has been revised and has been accepted for publication in the new Refractories Manual. Editorial revisions have been made for the new Manual on some of the other surveys already published.

Subcommittee II on Research (S. M. Phelps, chairman). A comparative study of methods of preparing cold-set mortar joints for determination of bond strength was made at the Refractories Fellowship at Mellon Institute. This information was used by Subcommittee VII in drawing up the new Tentative Specifications for Air-Setting Refractory Mortars (Wet Type) for Boiler and Incinerator Services (C 178 – 43 T).

Subcommittee III on Tests (F. A. Harvey, chairman) is composed of the chairmen of the several sections described below. The activities of the subcommittee have already been brought out in the foregoing discussion of recommendations accepted by Committee E-10, and are further amplified in the following discussion of the work of the individual sections:

Section B on Spalling (R. E. Birch, chairman) recommended tentative revisions of the panel test methods for spalling, Standards C 38 – 42, C 107 – 42, and C 122 – 42.

Section C on Temperature (R. B. Sosman, chairman) has made studies on the calcination of various types of ceramic materials prior to determining their P.C.E. This section also recommended tentative revisions in the Standard Method of Test for Pyrometric Cone Equivalent of Refractory Materials (C 24 – 42).

Section E on Analysis (L. J. Trostel, chairman) has been studying a method for the determination of ferrous iron in chrome refractory products. G. E. Seil of E. J. Lavino and Co. has presented a method after several years of study which is described in a recent paper by Mr. Seil.[3] Two representatives of the section have reported excellent results by the Seil method, and it will probably be considered by the section during the year looking toward its submittal for publication as tentative.

Section F on Tests on Refractory Insulation (W. R. Kerr, chairman) recommended editorial revisions in the Standard Methods of Testing Insulating Fire Brick (C 93 – 42) to bring the wording in line with the requirements of the Tentative Classification of Insulating Back-Up Block and Insulating Fire Brick (C 155 – 41 T), which have been accepted.

Section G on Porosity and Permanent Linear Change (C. E. Fulton, chairman) has been inactive during the past year.

Section H on Consistency and Plasticity (P. G. Herold, chairman) is making a study of the correlation of yield value and mobility determinations with the hope of gaining a fuller knowledge of plasticity and consistency of fireclay bodies.

A Bingham plastometer was constructed at the Missouri School of Mines, to be used in measuring plasticity in cold-set mortars.

A review of the literature on the subject of viscosity, plasticity, and consistency was presented by P. G. Herold and W. Smothers at the April, 1942, meeting of the American Ceramic Society. The manuscript is being revised and will be published in the *Journal* of the American Ceramic Society.

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[3] G. E. Seil, "Determination of Ferrous Iron in Difficultly Soluble Materials," *Industrial and Engineering Chemistry*, Analytical Edition, March 15, 1943, pp. 189–192.

A paper entitled "Control Testing for Consistency of Refractory Mortars" by E. C. Petrie and D. W. Kocher was published in the November, 1942, issue of the *Journal* of the American Ceramic Society.

Subcommittee IV on Heat Transfer (R. H. Heilman, chairman) has continued the cooperative work of comparison of thermal conductivity values on the same insulating fire brick in conductivity apparatus of the water calorimeter, calibrated plate, and hot plate designs. The Globar-heated calorimeter equipment developed by C. L. Norton was selected for use in the Tentative Method of Test for Thermal Conductivity of Insulating Refractories (C 182 - 43 T), prepared by the subcommittee.

Subcommittee V on Precision and Tolerance (R. A. Heindl, chairman) has been inactive during the year.

Subcommittee VI on Nomenclature (M. E. Holmes, chairman) believes that definitions which are useful and pertinent to the refractories industry are preferable to the dictionary type. The committee recommended a revised definition for "calcining" which has been accepted by Committee E-10 for publication as tentative.

Subcommittee VII on Specifications (G. A. Bole, chairman) prepared the Tentative Specifications for Air-Setting Refractory Mortar (Wet Type) for Boiler and Incinerator Services (C 178 - 43 T).

This subcommittee is considering specifications for the use of fire clays in service conditions other than that for laying up of fireclay refractories, which is covered in the Standard Specifications for Ground Fire Clay (C 105 - 41).

Subcommittee VIII, Editorial (L. J. Trostel, chairman) compiled a new edition of the Manual of A.S.T.M. Standards on Refractory Materials during the year. This includes specifications, classifications, methods of testing, definitions, recommended procedure for calculating heat losses through furnace walls, a list of standard samples of refractory materials for chemical analysis and for P.C.E. determinations, and industrial surveys of refractory service conditions in open-hearth practice, electric steel furnaces, malleable iron industry, copper industry, lead industry, by-product coke ovens, lime burning industry, glass industry, portland cement industry, and stationary steam boilers.

In addition to revisions mentioned earlier in this report, changes of an editorial nature were made in the Standard Methods for Chemical Analysis of Refractory Materials (C 18 - 41) and Test for Warpage of Refractory Brick and Tile (C 154 - 41).

Subcommittee IX on Classifications (S. M. Swain, chairman) is making a study to learn whether the present classification for insulating fire brick (C 155 - 41 T) can be improved.

This report has been submitted to letter ballot of the committee, which consists of 38 members; 34 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. D. SULLIVAN,
Chairman.
L. J. TROSTEL,
Vice-Chairman.

S. M. PHELPS,
Secretary.

APPENDIX

INDUSTRIAL SURVEY OF REFRACTORY SERVICE CONDITIONS IN ELECTRIC FURNACES USED IN STEEL MANUFACTURE

PART I. ARC FURNACES

Electric arc furnaces used in the production of steel may be divided into two classes from the standpoint of the refractories used and products produced. Basic steel is made in a furnace with a basic refractory hearth and acid steel in one with a siliceous type hearth.

The metallurgical functions for which the furnace is used may be divided into three general classes:

1. Melting only,
2. Melting and refining, and
3. Refining only.

The term "melting unit" is applied to a furnace in which melting is the chief requirement for conversion of the material, although some refinement usually occurs. A "melting and refining unit" is one in which the charge is melted and, in addition, is altered so that the product is of a different chemical composition and physical quality from the charge. The term "refining unit" is applied to a furnace which is charged with molten metal from an open hearth, bessemer converter, or a cupola, and in which the metal is changed chemically and physically. The furnace is sometimes used as a semicontinuous unit in which part of the metal is tapped at intervals and an equal tonnage charged after each pour.

The service life of the refractories varies according to the above classification of furnace use. This is because the time of heat, type of slag, and temperatures maintained will vary according to the original materials used, the chemical and metallurgical changes made in the furnace, and the composition and character of the product produced.

THE ARC PROCESS

Most direct arc furnaces are designed for three-phase, 60-cycle operation. Power transformers are required to convert the primary voltage into several secondary voltages in order properly to control the furnace. Automatic and manual equipment and instruments are required to control power input and to effect its economical use.

The path of the electric current for each phase is through one electrode, across the arc, through the slag and metal to another electrode, the circuit being then completed through the external circuit of the power transformer.

The voltages used and the arc developed greatly affect the refractories life. If the voltages are too high, the furnace lining is subjected to severe temperatures from the reflected arc and radiation fron the superheated slag and bath. Furnace design factors and the physical characteristics of the charge have much to do with the voltages used and the power input ranges necessary to obtain satisfactory operation.

DESIGN OF ELECTRIC-ARC FURNACES

Electric-arc furnaces range in capacity from ½ to 100 tons. In Table I, are given the capacity and size of representative commercial units. Figure 1 shows the cross-section of a typical design, and Table II presents the major dimensions for furnaces of various sizes. Except for the very largest sizes, the furnaces are circular in shape and use three electrodes. The larger the hearth area, the more difficult it is to obtain uniform temperature and to maintain and clean the hearth and banks. Because of this, and for other reasons connected with slag and metal control, the maximum size of a circular furnace is about 20 ft. in diameter. Larger units are elliptical in shape and require two groups of three electrodes.

Another classification affecting design is according to the method of charging, for example, door charging by hand, chute, or by a charging machine; and in the case of roof charging by a gantry crane or where the roof is either tilted or swung aside.

The service life of the refractories, especially in the roof, the door jambs, and the arches, is dependent upon the method of charging, because mechanical abuse by the charging apparatus varies according to the method used and the care exercised, and the severity of the thermal shock results from the time consumed in the charging operation.

TABLE I.—CAPACITY AND SIZE OF TYPICAL COMMERCIAL ELECTRIC-ARC FURNACES.

Inside Diameter of Shell	Nominal Charge, lb.	Transformer Capacity, kva.
7 ft. 3 in.	5 000 to 8 000	1 000 to 1 500
9 ft. 0 in.	10 000 to 12 000	2 000 to 3 000
10 ft. 0 in.	16 000 to 20 000	2 500 to 4 000
11 ft. 0 in.	20 000 to 24 000	3 000 to 4 500
12 ft. 0 in.	30 000 to 35 000	3 500 to 6 000
13 ft. 6 in.	36 000 to 40 000	5 000 to 7 500
15 ft. 6 in.	60 000 to 70 000	7 500 to 10 000
18 ft. 0 in.	100 000 to 130 000	10 000 to 12 000
19 ft. 0 in.	120 000 to 150 000	12 000 to 15 000
20 ft. 0 in.	160 000 to 170 000	12 000 to 18 000
6 electrode, elliptical shell 20 ft. by 29 ft.	200 000 to 230 000	20 000 to 24 000

All commercial arc furnaces are discharged (tapped) by tilting the furnace. Usually they can tilt forward approximately 48 deg. below the horizontal. In addition they are capable also of being tilted backward from 10 to 20 deg. to assist in slagging off, the slag being removed through the door opposite the tap hole.

Tilting permits the tap hole to be located above the slag level so it need not be packed shut as in the case of a stationary furnace, although this latter practice is sometimes used. This permits good maintenance of the tap hole and pouring spout. When tilting the furnace for tapping, however, the slag and steel level often reaches over the tap hole and comes in contact with the furnace side wall over the tap hole, so that the refractory in this location is subjected to severe erosive action.

Other design factors play important roles in the refractories life. The atmospheric volume contained within the furnace, as affected by the roof height and design of the side walls (vertical cylinder or inverted cone), determines the distance of the refractories from the arc and the concentration and distribution of the radiated heat on the refractories. A cone-shaped unit (a fairly new design) places the receding walls farther away from the concentrated heat of the arc. The angle of reflection is changed also so that the heat of the arc is said to be diffused to more surface area of refractory.

TABLE II.—MAJOR DIMENSIONS OF TYPICAL ELECTRIC-ARC FURNACES

Wall thickness varies according to individual plant practice, 4½ to 13½ in. in smaller furnaces, 12 to 18 in. in larger size units.

Nominal Capacity, tons	Diameter Inside Brick Walls	Bath Depth, Bottom to Fore Plate Level, in.	Height, Fore Plate to Base of Roof Skew
1½	4 ft. 11 in.	13½	2 ft. 9½ in.
5	7 ft. 6 in.	24	3 ft. 6 in.
10	8 ft. 9 in.	25	4 ft. 0 in.
20	12 ft. 0 in.	28	4 ft. 6 in.
35	13 ft. 6 in.	30	5 ft. 2 in.
60	16 ft. 9 in.	36	5 ft. 2 in.
80	17 ft. 9 in.	36	5 ft. 8 in.
100	Elliptical 17 ft. by 26 ft.	34	5 ft. 8 in.

The placement of the electrodes with respect to the furnace side walls affects refractory life. Generally the side wall adjacent to the masts holding the electrodes is the hottest part of the furnace, and the banks and linings here are subjected to the greatest erosion. Placement of the electrodes so that no electrode is adjacent to this side wall lessens the concentration of heat on this side of the furnace.

ELECTRODES

The electrodes used in direct arc furnaces are either the carbon or graphite type. They are manufactured from anthracite coal, petroleum coke or mixtures of these with a bond of some tar and hard and soft pitch.

The choice of the type of electrode used depends on such factors as economics, furnace design and kind of steel produced, because the amorphous carbon and graphite types each have their advantages and disadvantages. At furnace operating temperatures the electrical conductance of graphite electrodes is about twice that of the carbon, so they need not be as large in diameter. Being smaller they permit a stronger roof construction, lighter holding mechanism, lower transportation cost, and easier removal if they break and fall into the bath. They have no resistance losses at the joints and do not require electrode joint compound. Their resistance to oxidation is greater, which results in a consumption that is less than half that of the amorphous carbon electrodes.

Graphite electrodes, however, have the following disadvantages. They are more expensive per pound of electrode and are more fragile. Their higher heat conductivity results in more heat loss than with the amorphorous carbon type.

Graphite electrodes are used almost exclusively in the larger size furnaces, the carbon type being used for small electrodes because of their superior mechanical strength.

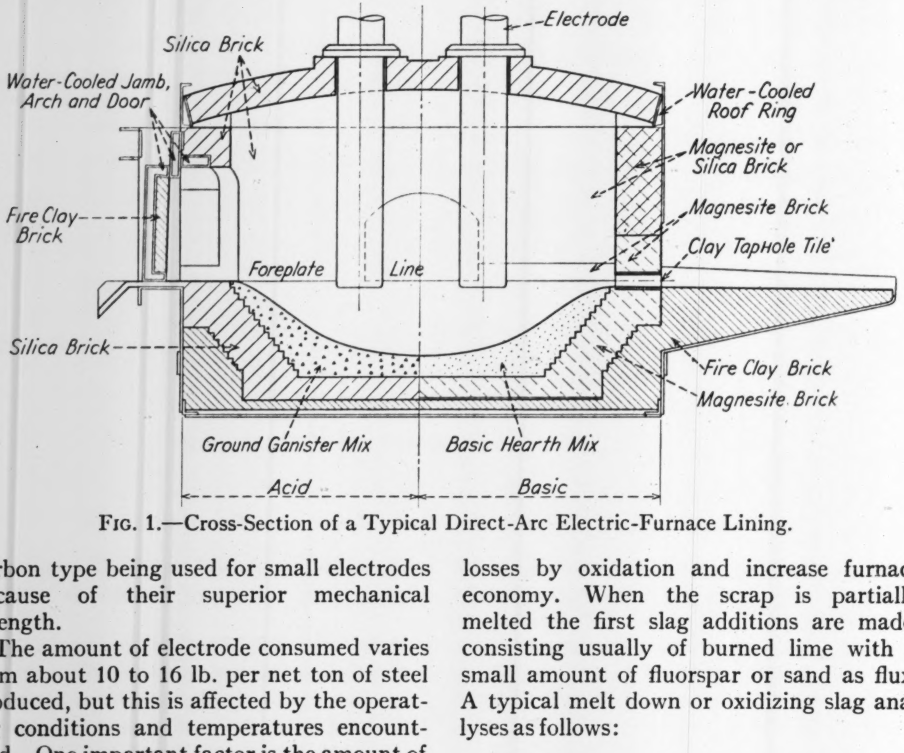
Electrode
Silica Brick
Water-Cooled Jamb, Arch and Door
Water-Cooled Roof Ring
Magnesite or Silica Brick
Fire Clay Brick
Magnesite Brick
Clay TapHole Tile
Foreplate Line
Silica Brick
Fire Clay Brick
Magnesite Brick
Ground Ganister Mix
Basic Hearth Mix
Acid
Basic

FIG. 1.—Cross-Section of a Typical Direct-Arc Electric-Furnace Lining.

The amount of electrode consumed varies from about 10 to 16 lb. per net ton of steel produced, but this is affected by the operating conditions and temperatures encountered. One important factor is the amount of electrode exposed inside the furnace as determined by the height of the roof over the bath.

THE BASIC ARC FURNACE

Basic Process:

In the basic process the heat is melted down under an oxidizing slag to remove phosphorus and also carbon, silicon, and manganese, as desired. The charge usually consists of steel scrap, alloys or alloy-containing scrap, and often a small amount of limestone. Considerable attention is paid to the physical condition of the scrap and its placement in the furnace in order that the heat can be properly melted.

At the start, intermediate voltages are applied to permit the electrodes to bore through the scrap. If the voltage is too high the molten metal may bridge over the heavy scrap on the bottom, resulting in a cold melt and skulled bottom. Later, a higher voltage is used in order to decrease losses by oxidation and increase furnace economy. When the scrap is partially melted the first slag additions are made, consisting usually of burned lime with a small amount of fluorspar or sand as flux. A typical melt down or oxidizing slag analyses as follows:

CaO, per cent	48.7
SiO_2, per cent	19.3
FeO, per cent	12.8
Fe_2O_3, per cent	3.25
MnO, per cent	6.50
MgO, per cent	7.10
P_2O_5, per cent	0.34
Al_2O_3, per cent	1.50
Cr_2O_3, per cent	0.30
S, per cent	0.19

The mineralogical constitution of the cooled slag is beta-dicalcium silicate, a solid solution phase of magnesium, manganese and ferrous oxides, undissolved magnesium oxide, magnetite (Fe_3O_4) and sometimes dicalcium ferrite and free lime. The phosphorus is probably present as tricalcium phosphate and in solid solution in the dicalcium silicate.

The heat can be finished by one of three slag practices:

1. Under the original oxidizing slag similar to open-hearth practice.
2. Making the original oxidizing slag reducing by the addition of ferrosilicon which reduces the iron oxide content with the resultant formation of silica. Any oxidizable alloying elements are also reduced to the metal bath. This slag change introduces additional reactions on the furnace refractories.
3. The two-slag practice, which is that most generally used. The oxidizing slag is removed by tilting the furnace backwards and removing the slag through the charging door. A reducing slag consisting of lime, silica, fluorspar, coke or ferrosilicon is added and the heat finished under this slag, which is effective in desulfurizing and deoxidizing the steel. The composition of carbide or reducing slag is:

CaO, per cent	61.5
SiO_2, per cent	24.5
C, per cent	0.67
CaC_2, per cent	3.54
CaF_2, per cent	1.80
FeO, per cent	0.43
Fe_2O_3, per cent	nil
MnO, per cent	0.09
MgO, per cent	4.34
Al_2O_3, per cent	2.76
S, per cent	0.41

Petrographic analysis of such a slag after cooling indicates gamma dicalcium silicate and usually tricalcium silicate, a solid solution phase of magnesium, manganese and ferrous oxide, magnesium oxide and sometimes free lime. Calcium carbide is known to be present although it cannot be seen under the microscope.

In this slag the unstable beta dicalcium silicate formed at high temperatures reverts to the stable gamma form on cooling with a subsequent volume increase of about 10 per cent. If this slag enters any cracks in the hearth of the furnace, it will expand upon cooling and will tend to rupture the bottom.

Modifications of the three practices given above are common and the slag composition will vary accordingly. In general, electric furnace slags are more fluid, and therefore more corrosive, than open hearth slags.

Refractories:

Bottoms.—The right-hand half of Fig. 1 shows the normal construction of the bottom of a basic arc furnace. The refractories consist of fireclay brick next to the furnace shell and then the magnesite brick, upon which is placed the basic grain bottom material. Infrequently there is used a buffer course of chrome brick or chrome plastic between the clay and the magnesite brick. The fireclay brick are used to produce a steeper temperature gradient through the bottom than would basic brick and they provide mechanical strength and stability. They are used around the shell sides, as a fill to reduce the cost below that incurred through the use of magnesite brick. An inch or more of an insulating refractory cement is often placed directly on the shell bottom before the clay brick are laid. The buffer course of chrome material is not used very often because the temperature at this point usually is not sufficient to promote reaction between the clay and magnesite bricks. The buffer material may be effective, however, in retarding slag or metal penetration through the bottom.

Magnesite brick are generally used in the next zone. The brick are laid carefully, with the use of keys and other standard shapes to obtain a sound structure. The banks or sides of the bottom are generally corbelled back to make proper contour and to provide a good base for the grain bottom.

The grain materials commonly used for the bottom are dead-burned magnesite and dolomite. Before installing in the furnace, a bonding or sintering material is added to the grain. The types and amounts of bond added vary considerably, depending upon the kind of grain used and the practices developed at individual steel plants, as will be described below.

The grain bottom is installed either by burning or sintering it into place in successive thin layers, or by tamping in a premixed composition. With the "burned in" type of bottom the grain material is mixed with 10 to 25 per cent of bond, usually finely ground open-hearth slag. Mill scale, iron ore, mica, or talc are sometimes used for the bond and these are generally in addition to the slag. Thin layers of the grain-bond mixture, approximately ½ to 1 in. in thickness are burned in while the furnace is at operating temperature.

Heat for burning in the bottom is obtained by placing scrap electrodes in a T formation on the bottom, which serves as a conductive medium for the carbon arc when the electrodes are lowered.

Materials for tamped-in bottoms usually have a hydraulic or oil type bond material in addition to the ceramic bonds mentioned above, so that it can be readily tamped and held in place.

Half-inch thick layers of the grain are tamped in with air hammers so that a dense monolithic bottom results. Wooden forms are sometimes used so that the proper thickness and contour can be obtained. The advantages that are claimed for the tamping method are better contour of the bottom, and less labor, time, and fuel input.

The thickness of the grain bottom varies according to the size of the furnace and the individual plant practice. Some bottoms are from 8 to 20 in. thick, while in other cases only a thin veneer of grain material from 1 to 4 in. is used. In the latter case a greater thickness of magnesite brick is used under the grain. The thin veneer naturally has a shorter life but it is more easily replaced, and because it is in the furnace for a shorter time, the bottom is cleaner with respect to metals which may be deposited in it and which later contaminate the steel.

Sintered dolomite is used for patching and maintaining the bottom, and this consists of "shaping up" the banks and filling in the cuts in the banks made by the slag. For large holes or major patches the regular bottom material may be used and sometimes is "burned in."

The furnace bottom must withstand the penetration, corrosion, and erosion by the slag and metal. In addition to having the correct chemical composition, it must be dense and free from cracks and voids. The surface should be smooth to provide drainage and to obviate focal points for the start of bottom boils.

Side walls.—The side walls of basic electric arc furnaces are constructed with either silica or basic brick. Economics has much to do with the selection but in addition such operating factors as size of furnace, shape, types of steel produced, and the effect of contamination of the slag by the refractories must be given consideration. The basic brick in the bottom extend up to door sill level and on this the side wall is laid. When silica brick are used, a buffer course of chrome is sometimes used between the basic and silica brick. This practice is not universal because the chrome may contaminate the metal bath. The basic walls are laid with magnesite brick which may be fired, chemically bonded, or metal encased.

The walls are usually laid up dry, using cement only to keep the courses level. In many furnaces water cooling pipes are placed between the shell and lining. When silica walls are used, basic brick are usually built up over and around the tap hole because, when tapping, the slag and steel will come in contact with this area.

The life of the side walls is determined principally by their ability to withstand the temperature produced in the furnace by the electric arc. They are subjected to abrasion by the charge and the action of the charging apparatus. One of their chief requirements is that they be mechanically strong and tight so that they will not shift or fall in when the furnace is tilted. They are also subjected to some slag attack from the melting scrap and from the oxide and vapor-laden gases produced in the furnace.

Roofs.—Silica, kyanite, or diaspore fireclay brick are used to construct the dome-shaped roofs. Silica brick are most generally used, and on the largest size furnaces they are used almost universally because of their high hot load strength.

On medium and small size melting units kyanite or alumina-diaspore refractories can be used to take advantage of their superior resistance to spalling. It has been reported recently that they are being used successfully and economically on units of over 50 tons capacity. The high spalling resistance afforded by this type of refractory is of special benefit when the roof is removed for charging, or when the furnace is operated intermittently. Monolithic roofs made of the kyanite or mullite types of ramming mixes have been used successfully on small furnaces where they are subjected to very high temperatures or intermittent operations.

TABLE III.—CLASSIFICATION OF REFRACTORIES FOR ELECTRIC-ARC STEEL FURNACES.

Furnace Part	Material		Temperature[a]	Slagging Action	Abrasion	Load	Spalling	Remarks
	Basic Type Furnace	Acid Type Furnace						
Hearth bottom and banks	Magnesite types of grain or ramming mixtures, dolomite.	Silica sand or ganister grain	2650 to 3100 F. (1455 to 1705 C.)	Severe	Severe	Unimportant	No	Infiltration of slag or metal into cracks may cause disruption of bottom.
Side walls	Silica brick; magnesite brick; burned, chemically bonded, or metal encased.	Silica brick	2650 to 3100 F. (1455 to 1705 C.)	Moderate	Mechanical abrasion of charge may be severe	Unimportant	Severe	Tilting of furnace may cause shifting of walls.
Roof	Silica, kyanite, or alumina-diaspore brick. Kyanite or mullite ramming mixtures.	Same as for basic type furnace	2650[b] to 3100 F. (1455 to 1705 C.)	Severe around electrode holes	Unimportant	Arch Stresses	Severe	
Tap hole	Clay tile, basic brick.	Clay tile	2600 to 3100 F. (1425 to 1705 C.)	Severe	Unimportant	No	Severe	Erosion is severe.
Spout	Clay or basic brick, plastic chrome or magnesite mixes, loam, fire clay.	Clay brick, fireclay, loam	2600 to 3100 F. (1425 to 1705 C.)	Severe	Unimportant	No	Severe	Erosion is severe.

[a] The equivalent Centigrade temperatures given in parenthesis are to the nearest five degrees.
[b] Direct fusion may occur due to high temperature of electric arc.

The electric furnace roof wears out due to loss of hot load strength, spalling, melting due to high temperature, and fluxing. The most severe attack on the roof is at the center section around the electrode holes. The severe corrosion and erosion which occur here are due primarily to the fact that there is a positive pressure in the furnace and large amounts of gases carrying fluxing oxides leave the furnace through the openings around the electrodes.

The general practice is to use standard shapes in constructing the roofs, but special shapes are sometimes used to lay up small roofs or to build the sections around the electrode rings on larger units in order to form a strong roof and to minimize the amount of brick cutting required. In some cases the center section of the roof, which includes the electrode holes, is made thicker than the remainder because it is subjected to more wear at this point.

All roofs are removable, because roof life usually is less than that obtained on the side walls. One to three roofs may be required with a single silica side wall lining, and, when basic linings are used, as many as ten roofs may be used with one lining.

The roof is held in a steel channel or angle-shaped ring against which the roof skew brick rest. In some newer designs a hollow water-cooled ring with a cross-section of a right angle triangle is used, in which case the roof brick are laid against the sloping side of the ring and no skew back brick are required.

Door Arches, Tap hole, and Spouts.—The arches over the doors are usually built of the same material as is used in the side walls. In door-charged furnaces these arches are subjected to extreme abrasion and mechanical shock from the charged scrap and the charging machine; consequently, they must be repaired frequently. Kyanite and alumina-diaspore refractories have been used successfully in this location, where their toughness and resistance to spalling may give them some advantage over silica or basic brick.

Two types of tap holes are used in electric arc furnaces: (*1*) An opening is left in the furnace wall just above the slag line in which is inserted a refractory clay tile. These tile are fairly dense and have Pyrometric Cone Equivalent of cone 27 to 30. The tile usually last for 2 to 6 heats and are replaced while the furnace is being charged. (*2*) The tap hole opening is below the slag-steel interface so that the furnace acts as a skimmer and holds back the slag until the steel has been discharged from the furnace. The "cartridge" type of tap hole is usually used in this case and it consists of a metal pipe into which has been packed the refractory grain sealing material. This cartridge is packed before being inserted in the side wall from the outside of the furnace. The space between the cartridge and the wall opening is then sealed from both outside and inside the furnace. On tapping, the packing is dug out from the outside, similar to the practice followed on open-hearths.

Some furnaces, generally small sized units, do not use a tap hole but have a small arched opening in the furnace wall. During operations a counterweighted door closes this opening. On tapping, the door is raised and slag and metal flow out simultaneously unless a skimmer is used to hold back the slag.

The spout or furnace runner may be lined with various materials, depending on individual plant preferences. Ladle brick are often used, as are magnesite, chrome, or fireclay brick. Occasionally loam or tamped granular materials of the same composition as the furnace bottom are used.

ACID ELECTRIC ARC FURNACES

Acid Process:

In the acid electric process the melt is made under a siliceous type of slag. With this process it is difficult to remove an appreciable quantity of phosphorus and sulfur; consequently, the raw materials used are kept comparatively low in these elements.

The acid process is used largely for making steel castings. Carbon removal and manganese control are more difficult in the acid than in the basic process and as a result the acid process is generally used only where high carbon and only moderate alloy contents are desired, such as is found in general foundry practice.

A typical acid slag has the following composition:

FeO, per cent	15
MnO, per cent	21
SiO_2, per cent	58
CaO, per cent	4
plus Al_2O_3, Cr_2O_3, etc.	

The percentage of FeO plus MnO is automatically regulated by the fact that iron manganese silicates will dissolve up to possibly above 60 per cent SiO_2 from the furnace banks. By regulating the amount of MnO in the slag the FeO can be closely controlled.

Refractories:

The refractories used in acid-lined electric arc furnaces have longer service life than those used in basic-lined furnaces, principally because the slag is more compatible with the refractories and also because the temperatures are generally somewhat lower.

The general operating and service conditions are as given above for the basic-lined units. All the refractories used are of the acid type. Roofs and side walls are constructed of silica brick. In the bottom as is shown in Fig. 1 clay brick are laid next to the steel shell and the silica brick which are laid on these are offset or corbelled on the sides to provide a suitable base for the grain material. The silica grain material may be installed either by sintering it into place in successive thin layers of about ½ to 1 in. thick, or by ramming. A rammed-in bottom usually consists of a mixture of 60 to 80 per cent crushed ganister and 20 to 40 per cent of finely divided, highly siliceous ramming refractory material of fairly high bond strength. The mixture is dampened with water and is rammed in solidly with air hammers. After drying, the furnace is charged and the melting started. Users of rammed silica bottoms claim a better bottom is obtained and at lower cost than is the case with sintered bottoms.

In sintered bottoms, a bond material such as lime, clay or acid slag is mixed with the grain. It is important that the proper type of sand or ganister is selected. If it is very high in silica, difficulty may be experienced in fritting it on the bottom, while if it contains too many impurities, it will frit easily but will be rapidly worn away when the furnace is put in operation. Chemical analysis of a sand cannot be used by itself as a criterion of its suitability for bottoms. Selection of a satisfactory material is also dependent upon the relative amounts of the impurities present and the physical characteristics of the grains. A typical bottom sand shows the following composition:

SiO, per cent	95.8
FeO, per cent	0.4
Al_2O_3, per cent	2.6
MgO, per cent	0.2
Ignition loss, per cent	0.8

The silica bottom must be sintered properly or there will be a tendency for the bottom material to "float up." Special attention is given to the first series of melts made on a new bottom so as to be sure that it is well matured.

PART II. THE CORELESS INDUCTION FURNACE

The coreless induction furnace may be of either the acid or basic type. It is used generally in the production of special or high-grade steel and castings, such as stainless steels, extremely low-carbon alloy steels, high-manganese steels, tool steels, and other highly alloyed types.

The cost of making steel by this furnace is said to be lower than that of the crucible process and in addition it produces greater homogeneity due to the constant stirring of the bath by the high motor effect of the current. The danger of carbon pickup is also eliminated. While the induction furnace has a higher cost per ton than the arc furnace, the electrical efficiency of the furnace itself is higher. The time of holding melts is shorter than in the arc furnace so that a 600-lb. induction unit can produce about the same tonnage in a day as a 4-ton arc furnace. Large amounts of power can be concentrated in a relatively small charge, so that rapid melting is achieved. From the thermal standpoint the refractories do not receive excessive punishment because it is not necessary to superheat them to melt the metal. The power input is under close control so the metal can be melted slowly or rapidly and temperature can be closely controlled.

THE INDUCTION PROCESS

The coreless induction furnace is essentially an air core transformer, the primary of which is the furnace coil and whose secondary is the melt. Figure 2 shows the essential parts of the furnace. The primary is a helical coil of copper tubing, insulated between turns, through which water is passed for cooling the coil. High-frequency current is applied to the coil and all the space inside the coil is subjected to a rapidly alternating electro-magnetic field. The furnace charge is inside the coil and is rapidly heated by reason of the current induced in it.

The frequency of the current varies according to the size and design of the furnace and, to some extent, according to the product being made. Large commercial units normally use frequencies ranging from 900 to 2000 cycles, while a small furnace producing metallic carbides may use a frequency as high as 20,000 cycles per second.

It is very difficult to refine with slag in the induction furnace and it is therefore not generally practiced. The composition of the product is controlled closely by knowing the exact composition of the charge and making allowances or corrective additions to compensate for the losses which occur. Such losses can be calculated in advance because they are relatively constant for a given furnace.

Vacuum refining is sometimes practiced in the induction furnace. In this process the furnace is equipped with a special top or lid which is sealed to the furnace. A line from the top is connected to a vacuum pump which draws off the gases evolved from the steel. Elimination of these gases produces a sounder ingot having better physical structure and fewer internal cracks or flaws.

DESIGNS

Induction furnaces are of cylindrical shape with the height of the metal bath approximately equal to the diameter. They are of the nose tilting type and are built in sizes ranging from 2 to 16,000 lb. The commercial sizes are usually 600 lb. and up, with melting cycles of one to three hours, depending on the power applied. The furnaces are top charged, this being done by hand in the smaller sizes and by charging buckets in the larger sizes.

Table IV presents the typical sizes, production rates, etc., of induction furnace units. Further details of design will be discussed below.

REFRACTORIES

Method of Application:

Furnaces up to 650 lb. capacity are usually lined with a prefired crucible. The coil is first covered with a $\frac{1}{4}$ to $\frac{3}{8}$ in. coating of a good troweling refractory mortar of the kyanite or mullite type. This coating protects the coil from being damaged by molten metal in case of a lining failure. Furthermore, this coating has a smooth surface and it is applied so as to have a slightly increasing diameter toward the top, which allows the lining to grow or move upward without catching or clinging to the coil.

The crucible is then set on a bed of the granular refractory (see Fig. 2) and the space between it and the coil is filled and tamped with the dry granular refractory almost to the top of the crucible. The top part is then sealed up with a refractory air setting mortar in which the pouring lip is formed. The crucibles sometimes crack, often during the first heat, but if there is a good rammed lining between the crucible and the coil, this does no particular damage and good service may still be attained.

In large furnaces the lining is tamped around a form which is usually tapered and is made of wood, steel, or asbestos lumber. A bed of fine granular refractory is placed in the bottom upon which the form is set. Granular refractory is tamped tightly between the coil and the form. The top is sealed and the pouring lip formed with refractory mortar as in the case with crucibles. The form is then removed and the lining thoroughly dried. A charge is then melted down, causing the lining to be sintered in place. When an asbestos form is used it frequently is left in place and when the first charge is melted it floats to the

TABLE IV.—SIZE AND PERFORMANCE OF TYPICAL INDUCTION FURNACES.

Furnace Size, lb.	Generator Size, kw.	Steel Melting Performance		
		Approximate Time to Melt	Approximate kw-hr. per ton over-all	Approximate lb. per hr.
50	60	20 min.	850	150
100	60	40 min.	850	150
150	60	1 hr.	850	150
200	60	1 hr. 20 min.	850	150
100	100	20 min.	800	300
200	100	35 min.	800	300
300	100	1 hr.	800	300
500	100	1 hr. 40 min.	825	300
300	150	35 min.	750	500
600	150	1 hr. 15 min.	750	500
1 000	150	2 hr. 15 min.	775	450
600	300	35 min.	700	1 000
1 000	300	1 hr.	700	1 000
2 000	300	2 hr. 15 min.	725	900
1 000	600	30 min.	675	2 000
2 000	600	1 hr.	675	2 000
4 000	600	2 hr. 15 min.	700	1 800
2 000	1 200	30 min.	650	4 000
4 000	1 200	1 hr.	650	4 000
8 000	1 200	2 hr. 15 min.	675	3 500
12 000	1 750	2 hr. 30 min.	675	4 800
16 000	Larger	(Have been built and placed in service recently, data not available)		

top of the bath as a slag and is then removed. Metal forms also may be left in place and in the first heat become part of the melt. During the melting some reaction occurs between the form and the lining material which aids in producing adequate sintering.

prefired crucibles are not used, the lining being tamped against a form as previously mentioned. The material may be tamped dry or a small amount of binder such as clay, boric acid, sodium silicate, or foundry compound may be added to provide proper ramming conditions.

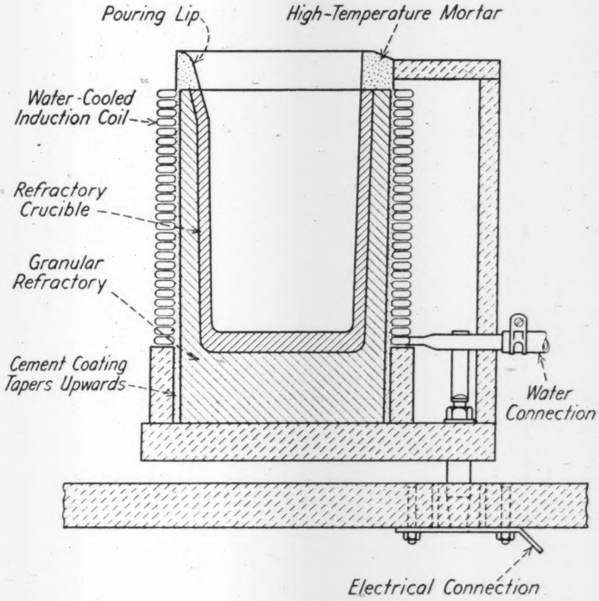

FIG. 2.—Cross-Section of Small Crucible Type Induction Furnace.

Basic Lining Materials:

When preformed crucibles are used they are usually made of clay bonded magnesite. The crucible is packed into the furnace with magnesite, zirconium silicate, or alumina grain. The packing material is usually a mixture of about 85 per cent of 60 mesh material and 15 per cent of fine flour and is tamped in dry. The grain size is of importance because the crucible tends to grow and expand and if held too tightly it may fail by cracking or by having the spout pull out. If the packing is too fine it may show a high firing shrinkage, be difficult to ram or if tamped too hard, it will cake and the crucible will not be able to adjust itself. When too coarse it will not sinter properly.

In furnaces of 650 lb. capacity or higher

Several types of materials are used, depending upon the kind of product being made and other operating considerations. Those commonly used are burned magnesite, a 70-30 ratio of fused magnesia and fused alumina (sometimes with about 5 per cent of clay as a binder), or 40 to 90 mesh zirconium silicate plus 15 per cent as a fine flour. In some cases a neutral lining of pure calcined alumina is preferred.

The sieve analysis of a typical fused magnesia and alumina lining mixture is:

Tyler Sieve Mesh No.	Per cent Retained	Tyler Sieve Mesh No.	Per cent Retained
3	0	28	7.1
4	1.9	35	4.2
6	5.4	48	5.3
8	7.2	65	5.2
10	8.8	100	4.5
14	14.1	150	4.9
20	14.1	200	10.5
		200	6.4 passing

Acid Lining Materials:

For acid linings a mixture of ganister and fine silica flour is used. Often a small amount of boric acid, sodium silicate or clay is added. The grain size analysis of a typical acid lining material is:

Tyler Sieve Mesh No.		Per cent
Passing	Retained on	
8	28	45
28	65	10
65	150	20
150	...	25

Preformed silica crucibles are used in small furnaces of laboratory size. Occasionally crucibles of siliceous fire clay are used in furnaces making certain grades of high-chromium steel or corrosion resisting alloys.

Refractories Service Conditions:

In addition to having sufficient refractoriness the linings must also be resistant to corrosion and erosion, have proper thermal and electrical properties and mechanical strength. For high electrical efficiency the refractory is kept as thin as possible. The lining insulates the coil from the charge both electrically and thermally. The selection of lining material sometimes is made in favor of the material having the better thermal insulating value.

Generally the lining wears out because of a combination of erosion and corrosion usually at the slag line. Although chemically active slags usually are not present in induction furnace practice, the stirring action of the melt produces a strong fluxing action at the level of the slag-metal interface and may cause the lining to fail at this point. Certain types of steel such as high-manganese steel, also cause excessive wear of the refractory. Furthermore, if proper charging and power application is not followed, part of the charge may tend to "bridge" and "hang up" in the upper part of the lining. The lining may be broken in attempting to break up this "bridge" or excessive temperatures may be produced in the bottom of the lining which may melt it out.

The rate of wear no doubt would be faster if it were not for the steep temperature gradient within the lining which tends to chill the fluxing materials as they penetrate. The rapidity of the heating also imposes a high thermal gradient on the refractory and after pouring the heat the lining tends to chill rapidly because the cooling water continues to pass through the coil.

Since the furnace is of the tilting type, the lining is subjected to mechanical strains imposed by the position and weight of the metal during the pouring operation. The charging operation also tends to produce mechanical shocks and abrasion. Similarly after pouring, the lining may be scraped with a metal tool to remove any adhering slag and if not done carefully this operation produces further abrasive and mechanical shocks.

As noted above, the rammed density and grain size of the lining demand proper control. Too high a proportion of fines or excessive ramming will tend to increase the depth of sintering. A sintered layer of about 1 in. thickness is usually satisfactory. Greater sintering increases conductivity and decreases resistance to shock. There must be enough sintering, however, to provide adequate strength.

Refractory Life:

The life of refractories in induction furnaces cannot be stated definitely. Size, design, type of product, and method of operation all play important roles. It can only be said that the linings of various commercial units may vary from less than 30 to more than 300 heats, depending on these conditions, the kind of refractory used and the care exercised in installing it.

REPORT OF COMMITTEE C-9

ON

CONCRETE AND CONCRETE AGGREGATES

Committee C-9 on Concrete and Concrete Aggregates has held no meetings since June 25, 1942. Ordinarily two meetings, one in the fall and one in the spring, are held each year in addition to the meeting during the annual meeting of the Society in June. The change this year was a direct outgrowth of the war. Members of the committee were too busy to give normal attention to committee work; it was desired to conform as much as feasible to Federal administration policy restricting travel; and projects before Committee C-9 did not involve critical materials nor did they appear to have an immediate bearing on the war effort.

It was with deepest regret that Committee C-9 learned of the death of Russell Greenman on March 15, 1943, at the age of 72 years. Mr. Greenman, a member of the Society since 1904, became a member of Committee C-9 in 1914. He was active in the work of the committee and was a faithful attendant at meetings. He was also active in other committees of the Society; he became a member of Committee D-4 on Road and Paving Materials in 1910 and of Committee C-1 on Cement in 1914, having been chairman of the latter committee for ten years beginning with 1916.

Mr. Greenman received his technical education at Union College, Schenectady, N. Y. In 1900 he was appointed Head of the testing laboratory of the Engineer's Department of New York. In 1927 he established a private practice specializing in consulting work on concrete. He was past-president of the Albany Society of Engineers, a director of the Albany County Chapter of the Society of Professional Engineers, and a member of the state and national organizations of that Society.

Subsequent to the 1942 annual meeting, Committee C-9 recommended to Committee E-10 on Standards that the five A.S.T.M. standards listed below which relate to curing of concrete be withdrawn inasmuch as these standards deal with a subject that, by agreement, is considered to fall within the scope of the American Concrete Institute rather than A.S.T.M. The A.C.I. has accepted jurisdiction over this general subject, having established Committee 612, Recommended Practice for Curing Concrete.

Standard Specifications for:

Curing Portland-Cement Concrete (C 80 – 34),

Standard Methods of:

Curing Portland-Cement Concrete Slabs with Bituminous Coverings (C 81 – 36),
Curing Portland-Cement Concrete with Calcium Chloride Admixture (C 82 – 38),
Curing Portland-Cement Concrete Slabs by Surface Application of Calcium Chloride (C 83 – 38), and
Curing Portland-Cement Concrete Slabs with Wet Coverings (C 84 – 36).

This recommendation was accepted by Committee E-10 on August 24, 1942, and the standards have accordingly been discontinued.

As a result of restricted activity, Committee C-9 has no new standards or revisions in standards to offer at this time. There are a number of tentative standards under the jurisdiction of the committee, some of which, under normal circumstances, undoubtedly, would be recommended for adoption as standard; on the other hand, several of them, even some of the older ones, would be recommended for continuation as tentative for at least another year. Under existing circumstances, it is recommended that all tentative standards and tentative revisions of standards under the jurisdiction of the committee be continued in their present status for at least another year. These include the following:

Tentative Specifications for:

- Ready-Mixed Concrete (C 94 – 42 T),[1]
- Waterproof Paper for Curing Concrete (C 171 – 42 T),

Tentative Methods of Test for:

- Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 41 T),
- Soundness of Aggregates by Freezing and Thawing (C 137 – 38 T),
- Efficiency of Materials for Curing Concrete (C 156 – 40 T),
- Volume Change of Cement Mortar and Concrete (C 157 – 40 T),[2]
- Sampling Fresh Concrete (C 172 – 42 T),
- Air Content of Freshly Mixed Concrete (C 173 – 42 T),
- Measuring Length of Drilled Concrete Cores (C 174 – 42 T),

Tentative Definitions of:

- Terms Relating to Concrete and Concrete Aggregates (C 125 – 42 T), and

Tentative Revision in Standard Method of:

- Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 39).

Action taken at the meeting planned to be held on July 1, 1943, in connection with the annual meeting of the Society, may result in recommendations making necessary an addendum to this report.[2] This report is accordingly restricted to brief references to the status of the committee's work.

Significance of Tests.—The revised Report on Significance of Tests of Concrete and Concrete Aggregates, mentioned in the 1942 annual report as nearing completion, has now been published. Comments on it are solicited, such comments to be retained in the files of the committee for consideration at some time in the future when it seems appropriate to make another revision. Also, the Society has made the ASTM BULLETIN available for publication of pertinent discussion of current interest.

Compilation of Standards.—The Compilation of A.S.T.M. Standards on Mineral Aggregates, sponsored by Committee C-9 and Committee D-4 on Road and Paving Materials, was issued in revised form during the year. While most of the standards included emanate from the sponsoring committees, the work of other committees has contributed to the compilation. The book includes methods of sampling and testing as well as specifications and definitions.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee V, Editorial and Definitions (C. M. Chapman, chairman).—This subcommittee has under consideration definitions of terms applying to concrete such as "curing," "workability," "water segregation," "bleeding," "water gain," "permeability," etc. Proposed definitions for most of the terms have been prepared but they are not as yet ready for submittal.

Subcommittee VI on Relation of Characteristics of Materials and Mixtures to Properties of Concrete (H. J. Gilkey, chairman).—The assignments of this subcommittee are less tangible than are those of subcommittees sponsoring standards. They depend largely on research and on the correlation of data from research. The subcommittee has not been active during the year although work has been going forward in several organizations, the results of which it will study. An important assignment is that of keeping in touch with the development of information pertaining to chemical reactions between aggregates and cement.

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[1] See Editorial Note, p. 244.
[2] See Addendum to Report, p. 245.

Subcommittee VII on Methods and Apparatus for Testing Concrete (A. T. Goldbeck, chairman).—This subcommittee has a number of important projects before it. It is developing information looking toward the preparation of standards for: determining water gain; making concrete test specimens from vibrated concrete; rate of loading of concrete test specimens; tests for uniformity of concrete proportions; making concrete beams in the field; methods of capping compression specimens; and freezing-and-thawing tests of concrete. It also proposes to make a critical review of all standards under its jurisdiction before the publication of the next edition of the Book of A.S.T.M. Standards.

Subcommittee VIII on Permeability of Concrete.—This subcommittee has been without a chairman since the death of Walter M. Dunagan on November 24, 1941. Prior to that time the subcommittee had been particularly active in developing papers on the general subject of permeability. The Advisory Committee has refrained, for the time being, from appointing a successor to Prof. Dunagan.

Subcommittee IX on Specifications and Methods of Tests of Aggregates (H. F. Clemmer, chairman).—This subcommittee is responsible for many of the specifications and test methods under the jurisdiction of Committee C-9. It has a number of projects before it, although no active work has been done on them during the year.

It now has under consideration further revisions in the Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 - 41 T) and in the Standare Method of Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 - 39). The latter revision has to do with clarifying requirements with respect to weight of sample. Study is being given to a major revision in the Standard Method of Test for Structural Strength of Fine Aggregate Using Constant Water-Cement-Ratio Mortar (C 87 - 42). As a basis for that study, mortar tests were conducted and reported by ten different laboratories. A report of these tests was prepared by a special subcommittee and definite recommendations made for changes in Standard Method C 87. A principal feature of these recommendations is to bring Method C 87 more closely in line with the Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 - 37 T) so that the mortar used for testing cement may also be used as a standard of comparison in testing sands.

A study of the applicability of the Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 - 39) to slag aggregates has been made by a member of the subcommittee and the results will be reported in a paper by Fred Hubbard and H. T. Williams, "Strength of Concrete as Related to Abrasion of the Blast Furnace Slag Used as Coarse Aggregate,"[3] to be presented at the annual meeting.

Work is also being done in the development of a revised method of test for surface moisture of fine aggregate which when completed will be recommended to replace Standard C 70 - 30, which has the same title.

Subcommittee XI on Evaluation of Data (H. W. Leavitt, chairman).—The principal project before this subcommittee is to cooperate with Technical Committee IX on Interpretation and Presentation of Data, of Committee E-1 on Methods of Testing, with a view to presenting working examples of the application of statistical methods to the evaluation of data on tests of concrete.

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[3] See p. 1088.

Subcommittee XIII on Curing of Concrete (W. F. Kellermann, chairman).—The work of this subcommittee during the past year has been confined largely to correspondence concerning proposed revisions in the Tentative Method of Test for Efficiency of Materials for Curing Concrete (C 156 - 40 T). Because of possible revision, it is recommended that the method be continued as tentative. The one project assigned to this subcommittee is entitled "A Study of What Constitutes Satisfactory Curing for Concrete."

Subcommittee XIV on Workability of Concrete (F. R. McMillan, chairman).—This subcommittee has been inactive during the year. However, it considers as its principal function that of keeping in touch with and encouraging the development of data on workability of concrete. It does not consider that the time has arrived when it is prepared to think in terms of specific specifications and test methods.

Subcommittee XVI on Elasticity and Volume Changes of Concrete (L. W. Teller, chairman).—The subcommittee has held no meetings during the year but has attempted to conduct a certain amount of work by correspondence. In 1940 it sponsored the Method of Test for Volume Change of Cement Mortar and Concrete (C 157 - 40 T). The subcommittee has revised this method somewhat and will recommend to Committee C-9 at its next meeting that it be adopted as standard as revised.

Subcommittee XVII on Conditions Affecting Durability of Concrete in Structures (C. H. Scholer, chairman).—This subcommittee has been inactive during the year. The status of the work on durability of concrete is such that little progress can be made during the war period. Methods of testing for durability are now changing very rapidly. New equipment is difficult to secure and trained personnel has, in general, been forced into more active participation in the war effort.

Subcommittee XVIII on Ready-Mixed Concrete (R. B. Young, chairman).—This subcommittee has held no meetings during the year but it has carried on considerable correspondence. It is making a careful review of the Tentative Specifications for Ready-Mixed Concrete (C 94 - 42 T)[1] and is giving careful study to a number of criticisms which have been offered of those specifications during the year. Particular attention is being paid to the development of a procedure for evaluating the concordance of acceptance tests. A report will be available for the consideration of Committee C-9 at its meeting in June.

This report has been submitted to letter ballot of the committee which consists of 68 members; 66 members returned their ballots, of whom 65 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

F. H. JACKSON,
Chairman.

STANTON WALKER,
Secretary.

[For Addendum to Report of Committee C-9, see p. 245.—Ed.]

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-9 presented to the Society through Committee E-10 on Standards a proposed revision of the Tentative Specifications for Ready-Mixed Concrete (C 94 - 42 T). The revised specifications were accepted by Committee E-10 on October 25, 1943, and assigned the designation C 94 - 43 T. They appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part II, p. 105.

ADDENDUM TO REPORT OF COMMITTEE C-9

At a meeting of Committee C-9 held on July 1, 1943, in Atlantic City, N. J., action was taken recommending the adoption as standard of the Tentative Method of Test for Volume Change of Cement Mortar and Concrete (C 157 - 40 T), with the following revisions, subject to approval by letter ballot of the committee:

Section 2.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of those in brackets:

2. (*a*) Molds for Mortar *and Concrete* Specimens.—Molds for casting *the* prismatic test specimens [of cement mortar] shall be made of metal and so [constructed] *designed* that the specimen [may] *will* be cast *with its longitudinal axis* in a horizontal position. The molds shall be fitted with metal end plates that provide means for holding a stainless steel [contact] *gage* point (Note 1) in exact position at the center of each end of the specimen during the placing and hardening of the *cement mortar or* concrete. Details of a suitable mold (*designed for mortar specimens*) are shown in Fig. 1.

NOTE 1.—The device for holding the contact points shall be arranged so that it can be *partially* released [slightly] after the mixture has been compacted into place in order to avoid restraint of the contact points during initial shrinkage of the specimen.

Delete the present Paragraph (*b*) and reletter the subsequent paragraph accordingly.

Figure 2.—Delete Fig. 2 showing the mold for horizontal casting of cylindrical concrete specimens and renumber the subsequent figure accordingly, and change the reference in Note 2 to read "Fig. 2" instead of "Fig. 3."

Section 3.—Change to read as follows by the addition of italicized words and the omission of the words in brackets:

3. (*a*) Mortar Specimens.—The test specimen for cement mortar shall be a prism 2 in. square and 11 in. in length [and shall be cast with the longitudinal axis of the prism in a horizontal position].

(*b*) Concrete Specimens.—The test specimen for concrete shall be a [cylinder] *prism* 3 in. [in diameter] *square* and 11 in. in length. [The maximum size of aggregate in the concrete for such a specimen shall not exceed ¾ in. If desired, the cylindrical concrete specimens may be cast with the longitudinal axis of the cylinder in a horizontal position, provided a mold of suitable design is used.]

(*c*) Contact *or* Gage Points.—Each test specimen shall have embedded at each end of the longitudinal axis, a stainless steel contact *or gage* point [or surface] so designed as to provide an effective [shrinkage] *gage* length of 10 ± 0.01 in.

Section 4 (*b*).—Change from its present form: namely,

(*b*) All materials shall be measured and proportioned by weight. The measuring and mixing of the materials and the placing of the concrete in the molds shall be performed as described in the Standard Method of Test for Compressive Strength of Concrete (A.S.T.M. Designation: C 39) of the American Society for Testing Materials. Specimens of cement mortar shall be molded by the same procedure as prescribed for concrete specimens whenever possible.

to read as follows:

(*b*) All materials shall be proportioned by weight. The mixing and molding procedure shall follow, as nearly as possible, those prescribed in the Standard Method of Test for Flexural Strength of Concrete (A.S.T.M. Designation: C 78) of the American Society for Testing Materials.

Section 5.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

5. (*a*) Immediately after molding, the test specimens in the molds shall be covered with wet burlap or placed in the *moist* [storage] room.

After 20 to 24 hr. the specimens shall be removed from the molds and placed in the storage room.

NOTE.—In the case of certain slow-hardening cements it may be necessary to allow specimens to remain in the mold for more than 24 hr. in order to avoid damage during removal from the molds. When making tests in which such cements are included, the storage schedule may be modified, but *any* [all] specimens involved in the test program, *which are to be used for comparison,* shall be subjected to identical conditions of storage.

(*b*) The storage room shall be maintained at a temperature of *70 ± 2 F.* [80 ± 2 F.] and a relative humidity of 50 ± 5 per cent.

This recommendation was submitted to letter ballot of the committee which consists of 75 members; 62 members returned their ballots, of whom 57 have voted affirmatively, 4 negatively, and 1 member marked his ballot "not voting."

Respectfully submitted on behalf of the committee,

F. H. JACKSON,
Chairman.

STANTON WALKER,
Secretary.

REPORT OF COMMITTEE C-14
ON
GLASS AND GLASS PRODUCTS

Committee C-14 on Glass and Glass Products held one meeting since presenting its 1941 report, on April 21, 1943, in Pittsburgh, Pa.

As indicated later in this report, two new subcommittees have been formed, namely, Subcommittee VII on Glass Containers and Subcommittee VIII on Flat Glass.

Subsequent to the 1941 Annual Meeting, Committee C-14 presented to the Society through Committee E-10 on Standards new Tentative Methods of Chemical Analysis of Soda-Lime Glass. These methods were accepted[1] by Committee E-10 for publication as tentative on August 25, 1941, and appear in the 1942 Book of A.S.T.M. Standards, Part II, bearing the A.S.T.M. serial designation C 169 - 41 T.

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative standards be approved for reference to letter ballot of the Society for adoption as standard and that they be revised as referred to in the reports of the responsible subcommittees:

Tentative Methods of:[2]

Chemical Analysis of Glass Sand (C 146 - 39 T), proposed by Subcommittee II,
Hydrostatic Pressure Test on Glass Containers (C 147 - 40 T), revised as proposed by Subcommittee IV,
Polariscopic Examination of Glass Containers (C 148 - 40 T), revised as proposed by Subcommittee IV,
Thermal Shock Test on Glass Containers (C 149 - 40 T), revised as proposed by Subcommittee IV,
Flexure Testing of Glass (Determination of Modulus of Rupture) (C 158 - 40 T), revised as proposed by Subcommittee IV, and
Chemical Analysis of Soda-Lime Glass (C 169 - 41 T), revised as proposed by Subcommittee II.

These recommendations have been submitted to letter ballot of the committee which consists of 40 members; 00 members returned their ballots with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Chemical Analysis of Glass Sand (C 146 - 39 T)	22	0	7
Hydrostatic Pressure Test on Glass Containers (C 147 - 40 T), as revised	20	0	9
Polariscopic Examination of Glass Containers (C 148 - 40 T), as revised	23	0	5
Thermal Shock Test on Glass Containers (C 149 - 40 T), as revised	20	0	9
Flexure Testing of Glass (Determination of Modulus of Rupture) (C 158 - 40 T), as revised	21	0	7
Chemical Analysis of Soda-Lime Glass (C 169 - 41 T), as revised	22	0	7

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (F. C. Flint, chairman).—On the recommendation of the Subcommittee on Definition of Glass of Committee E-8 on Nomenclature and Definitions, consisting of representatives of Committees C-1 on Cement, C-5 on Fire Tests of Materials and Construction, C-14 on Glass and Glass Products, C-15 on Manufactured Masonry Units, D-9 on Electrical Insulating Materials, D-13 on Textile Materials, and D-20 on Plastics, Subcommittee I is planning to review the Tentative Definition of the Term Glass (C 162 - 41 T) looking toward the development of a definition, or several definitions, to cover such terms as glass as a material, glasslike, glassy state, and possibly so-called "organic glass."

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee C-14 reported results of the letter ballot vote as follows: Of a total membership of 41, 33 members returned their ballots, of whom 26 voted affirmatively, 0 negatively, and 7 members marked their ballots "not voting."

[2] 1942 Book of A.S.T.M. Standards, Part II.

Subcommittee II on Chemical Analysis (G. E. F. Lundell, chairman).—Before the Tentative Methods of Chemical Analysis of Glass Sand (C 146 - 39 T) and of Soda-Lime Glass (C 169 - 41 T) were published in the 1942 Book of A.S.T.M. Standards, certain editorial changes were included. The subcommittee now recommends that Tentative Methods C 146 be adopted as standard without change and that Tentative Methods C 169 be adopted as standard, revised as follows:

Section 5 (*a*).—Add the following information to the tabulation with reference to the probable accuracy of results:

Constituent	Probable Accuracy of Results, per cent Referee Analysis	 Routine Analysis
CaO	±0.05	±0.05
MgO	±0.05	±0.05

Section 6 (*c*).—Change the first sentence from its present form: namely,

> Add 3 or 4 drops of diluted H_2SO_4 (1:1) and ignite the contents of the dish, slowly at first and finally for 30 min. to 1 hr. at 1200 C.

to read as follows:

> Ignite the contents of the dish slowly at first and finally at approximately 600 C. until the carbon is gone. Add 3 or 4 drops of diluted H_2SO_4 (1:1) if a high nonsilica residue is expected, and ignite slowly at first and finally for 30 min. to 1 hr. at 1200 C.

Section 20 (*d*).—Change the third sentence to read as follows by the addition of the italicized words:

> Add 5 drops of the methyl red solution (*paranitrophenol may be substituted if desired*) and then diluted HCl (1:1) drop by drop until the solution is acid, taking care not to add more than 1 drop in excess.

Section 23.—Add a new sentence after the fourteenth sentence of Paragraph (*a*), following the washing of the silicon dioxide precipitate, to read as follows:

> If desired the first dehydration can be made as described in the Procedures for Referee Analysis (Section 6 (*a*)); in this case add 10 to 15 ml. of $HClO_4$ (60 per cent) before the second evaporation.

Add a new sentence after the fifth sentence of Paragraph (*b*) to read as follows:

> Repeat ignition to constant weight.

Subcommittee III on Chemical Properties (F. W. Adams, chairman).—This subcommittee was organized to undertake work on the chemical properties of glass as glass. The trend is now toward the testing of glass containers. In order to take care of all these problems adequately, it has been recommended that the subcommittee limit its scope to the chemical properties of glass as glass, and that a new subcommittee be formed to consider the properties of glass containers.

Since Mr. Adams is closely connected with the flat glass industries, he has resigned from the chairmanship of Subcommittee III, and is succeeded by J. F. Greene. Mr. Adams has been appointed chairman of the new Subcommittee VIII on Flat Glass.

Subcommittee IV on Physical and Mechanical Properties (J. T. Littleton, chairman).—The subcommittee recommends that the following tentative methods be revised as indicated below and adopted as standard:

Tentative Method of Hydrostatic Pressure Test on Glass Containers (**C 147 - 40 T**):[2]

Section 3 (*a*).—Change the heading of this paragraph to read as follows by the omission of the words in brackets:

Sampling [from Lehr] in Manufacturing Plant During Continuous Production:

Replace the present Item (*2*) with the following new Items (*2*) and (*3*):

(*2*) For continuous routine checking of quality, including handling and packing, one bottle from each cavity or mold shall be taken every 3 hr. from cartons packed by the inspector or the packer and in such a manner that the packer does not know which cartons will be opened by the sampler.

(*3*) For a detailed test of new mold designs or of radical changes in machine operations a sample of 10 bottles from each mold or cavity shall be required for test. In case the total number of molds shall be more than 10, a total of 100 bottles distributed among the molds shall be required for the test. The bottles shall be taken from the exit end of the annealing lehr.

Tentative Method of Polariscopic Examination of Glass Containers (**C 148 - 40 T**):[2]

Section 1.—Change the first sentence to read as follows by the addition of the italicized word and the omission of the word in brackets:

This method for the polariscopic examination of glassware describes the procedure for determining the relative [internal] *annealing* stress in glass containers by comparison with reference standards.

Add a new sentence at the beginning of the Note following this section to read as follows:

The method is not intended to evaluate the cordiness or cord stresses due to composition inhomogeneities.

Section 3 (*a*).—Change the heading of this paragraph to read as follows by the omission of the words in brackets:

Sampling [from Lehr] in Manufacturing Plant During Continuous Production.

Section 4.—Change the Note following Paragraph (*a*) from its present form: namely,

NOTE.—The color observed by the procedure described in Paragraphs (*b*), (*c*), and (*d*) should be the broad color patterns produced by the lehr, and this temper is considered the "lehr temper."

to read as follows:

NOTE.—The color observed by the procedure described in Paragraphs (*a*), (*b*), and (*c*) should be the broad color patterns produced by annealing stresses, and this temper is considered the "lehr temper." Local stresses clearly due to cords or composition inhomogeneities should be distinguished from the broad pattern and ignored in the assignment of the temper grading.

Delete Paragraph (*d*) on examination of cordy glass which reads as follows:

(*d*) *Examination of Cordy Glass.*—When the most noticeable colors observed are streaks and irregular markings clearly due to cord, the glassware at these points shall be examined and the temper obtained designated as "cord temper" (see Note, Paragraph (*a*)).

Tentative Method of Thermal Shock Test on Glass Containers (**C 149 - 40 T**):[2]

Section 3 (*a*).—Make the same changes in this section as recommended above in Section 3 of Method C 147.

Tentative Method of Flexure Testing of Glass (Determination of Modulus of Rupture) (**C 158 - 40 T**):[2]

Section 6 (*a*).—In the explanation of terms under the formula, change the description of symbols b and d to read as follows by the omission of the words in brackets:

b = width of specimen [at rupture] in inches,

d = thickness of specimen [at rupture] in inches.

Appendix.—Change the second sentence of Paragraph A3 to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> This is equivalent respectively to [91.8 mμ and 183.6 mμ] *90 mμ and 180 mμ* per inch of light path in common glass.

Change the last sentence of Paragraph A4 to read as follows by the addition of the italicized word and figure and the omission of those in brackets:

> The stress shall not exceed 400 psi. (equivalent to [184 mμ] *180 mμ* per inch of length for common soda lime glass).

There has been some discussion in the subcommittee about the possibility of having work started on the thermal endurance of glass.

Subcommittee VII on Glass Containers (W. R. Lester, chairman).—This new subcommittee was formed to give consideration to glass containers in their many forms, as products.

Subcommittee VIII on Flat Glass (F. W. Adams, chairman).—This new subcommittee was formed to give consideration to flat glass from the viewpoint of products.

This report has been submitted to letter ballot of the committee which consists of 40 members; 33 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. C. HOSTETTER,
Chairman.

LOUIS NAVIAS,
Secretary.

REPORT OF COMMITTEE C-16
ON
THERMAL INSULATING MATERIALS

Committee C-16 on Thermal Insulating Materials has not met since its meeting held in Atlantic City, N. J., on June 25, 1942, during the Annual Meeting of the Society. Reduced personnel, resulting from the induction of younger men into the Armed Forces and the volunteering of older men for technical service in the Army and Navy, has so increased the regular work of the members of Committee C-16 that it was decided at the meeting held in Cleveland, Ohio, on March 5, 1942, to abandon the 1942 fall meeting, but to meet during the 1942 Annual Meeting of the Society. By unanimous vote of the Advisory Committee and the chairmen of the subcommittees of Committee C-16 it was decided not to call a meeting during the 1943 spring group meetings in Buffalo, N. Y. The committee is planning to meet during the 1943 Annual Meeting of the Society in Pittsburgh, Pa.

At the June, 1942, meeting of the Advisory Committee, the temporary organization of Committee C-16, as described in the 1942 report of the committee, was adopted with some modification. The present organization of the subcommittees has been published in the August, 1942, issue of the A.S.T.M. Year Book.

Since the last report, eight changes in membership have occurred. Three manufacturers, not previously represented, have nominated representatives and five changes in representation have been necessary because of the pressure of war activities. At present the committee consists of 53 voting members, 2 nonvoting, and 3 consulting members.

The committee regrets the loss through resignation of H. H. Rinehart, Vice-Chairman of the committee and chairman of Subcommittee III. Mr. Rinehart has done outstanding work on this committee.

The activities of Committee C-16 during the year culminated in the completion of the following 12 emergency standards:

Emergency Specifications for:

85 Per Cent Magnesia Thermal Insulating Cement (ES - 8),
Long-Fiber Asbestos Thermal Insulating Cement (ES - 9),
Mineral Wool Thermal Insulating Cement (ES - 10),
Expanded or Exfoliated Mica Thermal Insulating Cement (ES - 11),
Diatomaceous Silica Thermal Insulating Cement, for Use from 600 to 1200 F. (ES - 12),
Diatomaceous Silica Thermal Insulating Cement, for Use from 1200 to 1900 F. (ES - 13),
Blanket Thermal Insulation for Building Purposes (ES - 14),
Blanket Thermal Insulation for Industrial Purposes (ES - 15),
Blanket Thermal Insulation for Refrigeration Purposes (ES - 16),
Preformed Pipe Covering Thermal Insulation (ES - 17),
Preformed Block Thermal Insulation (ES - 18), and
Structural Board Insulation (Thermal Insulation) (ES - 19).

These emergency specifications were approved by Committee C-16 at its June, 1942, meeting and submitted to

Committee E-10 on Standards. They were accepted by Committee E-10 on August 24, 1942, and assigned the A.S.T.M. serial designations indicated above. They appear in the 1942 Book of A.S.T.M. Standards, Part II.

ACTIVITIES OF SUBCOMMITTEES

The present subcommittee structure of Committee C-16 is as follows:

Subcommittee I on Physical Properties of Preformed Block Insulation, Including Pipe Covering (R. H. Jebens, chairman).—This subcommittee assumed the work of Subgroups A and C, as reported in 1942, and is planning to redraft Emergency Specifications ES - 17 and ES - 18 into a form suitable for submittal to the Society for publication as tentative.

Subcommittee II on Physical Properties of Structural Insulating Board Made from Both Mineral and Vegetable Materials (Paul D. Close, chairman).—This subcommittee prepared Emergency Specifications ES - 19 and is charged with the responsibility of approving the test methods as now described in the specifications or devising new methods that would be applicable to the materials covered in these specifications. No action was taken regarding the Tentative Methods of Test for Compressive Strength and Flexural Strength of Preformed Block Type Thermal Insulating Materials (C 165 - 41 T) and the Tentative Definitions of Terms Relating to Thermal Insulating Materials (C 168 - 41 T). The subcommittee recommends that these two tentative standards be continued in their present status for another year.

Subcommittee III on Physical Properties of Thermal Insulating Cement (H. W. Greider, chairman).—Emergency Specifications ES - 8 to ES - 13, inclusive, were prepared by this subcommittee. It is now engaged in obtaining final approval of these specifications looking toward their submittal to the Society for publication as tentative. This action should be completed soon. The Tentative Methods of Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (C 166 - 41 T) will be continued as tentative for another year.

Subcommittee IV on Physical Properties of Blanket and Semirigid Insulation Made from Both Mineral and Vegetable Materials (R. E. Cryor, chairman).—Emergency Specifications ES - 14, ES - 15, and ES - 16 were prepared by this subcommittee. These three specifications will be reviewed and the test procedures required will be prepared.

Subcommittee V on Physical Properties of Loose-Fill Thermal Insulation Made from Both Mineral and Vegetable Materials (G. G. Christner, chairman).—This is a new subcommittee that was formed to execute a part of the work of the original Subcommittee IV. The enlargement of the scope of Committee C-16 to include building insulation made advisable the formation of this new group, thus lightening the load of the group that is now Subcommittee IV.

Subcommittee VI, the A.S.T.M. Group of the Joint Committee on Thermal Conductivity of All Forms of Insulation (F. C. Houghten, chairman).—During the past year the joint committee has not been active because of the absence of the chairman in active duty in the United States Navy and because of the abnormally large amount of work being carried on by many members of this group. The matters of the guarded hot box procedures for measuring heat transfer through assembled wall structures and that for measuring heat flow through preformed pipe covering will be taken up as soon as conditions approach normalcy.

Subcommittee VII on Measurement of Thermal Properties Other Than Thermal

Conductivity of Thermal Insulating Materials (C. B. Bradley, chairman).—This subcommittee, replacing original Subcommittee VII, was organized during the past year and will undertake to standardize methods for the determination of specific heat, coefficient of linear expansion, and other thermal properties of heat insulating materials.

Subcommittee VIII on Studies of Dimensional Standards of Preformed Block Insulation (Ray Thomas, chairman).—There have been no new developments in the work of this subcommittee since the report made a year ago except informal notice taken of its work by the War Production Board.

Subcommittee IX on Studies of Vapor Barriers Applied to Thermal Insulation Used Especially in the Air Conditioning and Refrigeration Fields (R. H. Heilman, chairman).—This is a new subcommittee that has been formed as a result of a demand coming from the users of building and refrigerator insulation. There appears to be great need for standardization of test procedures affecting vapor barriers.

Subcommittee X on Editorial Functions (B. A. Hollenbeck, chairman).—This subcommittee has, by correspondence, edited all of the Emergency Specifications and has largely prepared the descriptions of the three common types of guarded hot plate for use in connection with the Tentative Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (C 177 - 42 T). Copies of these detailed descriptions of the three guarded hot plates known as the National Bureau of Standards plate, the National Research Council plate, and the Alundum plate, are obtainable from A.S.T.M. Headquarters.

Subcommittee XI for Liaison with A.S.T.M. Committee C-8 on Refractories (R. H. Heilman, chairman).—Since no matters affecting both of the participating committees have arisen during the year, no meetings have been held.

This report has been submitted to letter ballot of the committee which consists of 53 members; 39 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

E. T. COPE,
Chairman.

RAY THOMAS,
Secretary.

REPORT OF COMMITTEE D-1

ON

PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: on June 23, 1942, in Atlantic City, N. J., during the annual meeting of the Society, and on March 2, 1943, in Buffalo, N. Y., during the spring group meetings of the Society.

At the March meeting, Committee D-1 arranged for the presentation of a talk by E. A. Tice, on "The Effect of the Degree of Weathering and Methods of Surface Pretreatment on the Primer Life of Structural Steel."

During the past year a new subcommittee has been appointed, Subcommittee V on Volatile Solvents for Organic Protective Coatings. This subcommittee will take over the activities of Subcommittee XII on Turpentine and Subcommittee XI on Paint Thinners Other than Turpentine which subcommittees have been discontinued.

Subcommittee XIII on Shellac has been established on an inactive basis with W. H. Gardner continuing as chairman.

H. E. Smith has been appointed representative of Committee D-1 on the Advisory Committee on Corrosion, with H. A. Nelson as alternate.

Subsequent to the annual meeting, Committee D-1 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:

Isopropyl Acetate (D 657 – 42 T),

New Tentative Methods of:

Evaluating Degree of Chalking of Exterior Paints of the Linseed-Oil Type (D 659 – 42 T),

Evaluating Degree of Checking of Exterior Paints of the Linseed-Oil Type (D 660 – 42 T),

Evaluating Degree of Cracking of Exterior Paints of the Linseed-Oil Type (D 661 – 42 T),

Evaluating Degree of Erosion of Exterior Paints of the Linseed-Oil Type (D 662 – 42 T),

Reversion of Standard to Tentative:

Standard Specifications for Basic Sulfate White Lead (D 82 – 41), as revised, replacing the Emergency Alternate Provisions EA - D 82 which were discontinued, and

Tentative Revision of Standard Specifications for:

Zinc Oxide (D 79 – 41).

These recommendations were accepted[1] by Committee E-10 on August 24, 1942, and assigned the A.S.T.M. serial designations indicated above. The new and revised tentative standards as well as the tentative revision appear in the 1942 Book of A.S.T.M. Standards, Part II.

On September 16, 1942, Committee E-10 accepted Emergency Alternate Provisions in the Standard Specifications for Mica Pigment (D 607 – 42) which were issued under the designation EA –

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-1 reported the following results of the letter ballot vote of a total of 123 ballots returned from a membership of 222: D 657, affirmative 75, negative 0, ballots marked "not voting" 48; D 659, affirmative 104, negative 1, ballots marked "not voting" 18; D 660, affirmative 103, negative 1, ballots marked "not voting" 19; D 661, affirmative 103, negative 1, ballots marked "not voting" 19; D 662, affirmative 103, negative 1, ballots marked "not voting" 19; D 82, affirmative 78, negative 3, ballots marked "not voting" 42; D 79, affirmative 88, negative 2, ballots marked "not voting" 33.

D 607. They were published in ASTM BULLETIN, No. 118, October, 1942, and have been issued in the form of a pink sticker for attachment to the standard.

Of the one adviser and 14 subcommittees, 13 furnished reports of progress and 6 submitted detailed reports affecting standards which have resulted in the committee making the recommendations given below to be acted upon at the annual meeting.

In this report, recommendations affecting standards and tentative standards are noted in brief form below, together with the results of the letter ballot. These recommendations are explained in the reports of the subcommittees directly responsible for them.

I. NEW TENTATIVE STANDARDS

The committee submits for publication as tentative the following nine methods of test:

Methods of Test for:[2]

Conducting Road Service Tests on Traffic Paints, proposed by Subcommittee IV,
Light Sensitivity of Traffic Paints, proposed by Subcommittee IV,
Dry-To-No-Pick-Up-Time of Traffic Paints, proposed by Subcommittee IV,
Evaluating the Degree of Blistering of Organic Coatings when Subjected to Immersion or Other Tests Involving Exposure to Moisture or Liquids, proposed by Subcommittee VII.
Analysis of Barium Sulfate Pigments, proposed by Subcommittee VIII,
Analysis of Mica Pigment, proposed by Subcommittee VIII,
Analysis of Magnesium Silicate Pigment, proposed by Subcommittee VIII,
Analysis of Aluminum Silicate Pigment, proposed by Subcommittee VIII, and
Analysis of Diatomaceous Silica Pigment, proposed by Subcommittee VIII,

II. REVISION OF TENTATIVE STANDARD

The committee recommends that the Tentative Method of Test for Phthalic Anhydride Content of Alkyd Resin Solutions (D 563 – 40 T)[3] be revised as referred to in the report of Subcommittee IX and continued as tentative.

III. REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption, revisions in the Standard Methods of Testing Oleoresinous Varnishes (D 154 – 38)[3] as proposed in the report of Subcommittee IX, and in the Standard Specifications for Basic Carbonate White Lead (D 81 – 41)[3] as proposed in the report of Subcommittee XV, and accordingly asks for a nine-tenths affirmative vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society.

IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative standards[3] be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Specifications for:

Chrome Yellow and Chrome Orange (D 211 – 42 T), proposed by Subcommittee XV,
Liquid Paint Driers (D 600 – 41 T), proposed by Subcommittee IX,
C. P. Toluidine Toner (D 656 – 42 T), proposed by Subcommittee XV,
Dibutylphthalate (D 608 – 41 T), proposed by Subcommittee XXV,

Tentative Methods of:

Testing Liquid Driers (D 564 – 41 T), proposed by Subcommittee IX, and
Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces (D 610 – 41 T), proposed by Subcommittee VII.

These recommendations have been submitted to letter ballot of the committee which consists of 231 voting members, with the results shown in Table I.

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[2] These methods were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part II, pp. 142, 140, 138, 144, 123, 132, 129, 119, and 127, respectively.

[3] 1942 Book of A.S.T.M. Standards, Part II.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Drying Oils (Francis Scofield, chairman).—This subcommittee recommends that the Tentative Specifications for Oiticica Oil (Permanent Liquid) (D 601 – 41 T) be continued in their present status without revision.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Test for Conducting Road Service Tests on Traffic Paints	36	0	47
Test for Light Sensitivity of Traffic Paints	47	0	36
Test for Dry-To-No-Pick-Up-Time of Traffic Paints	46	0	37
Test for Evaluating the Degree of Blistering of Organic Coatings When Subjected to Immersion or Other Tests Involving Exposure to Moisture or Liquids	52	0	31
Analysis of Barium Sulfate Pigments	46	1	36
Analysis of Mica Pigment	46	1	36
Analysis of Magnesium Silicate Pigment	48	0	35
Analysis of Aluminum Silicate Pigment	48	0	35
Analysis of Diatomaceous Silica Pigment	47	0	36
II. REVISION OF TENTATIVE STANDARD			
Test for Phthalic Anhydride Content of Alkyd Resin Solutions (D 563 – 40 T)	43	0	40
III. REVISION OF STANDARDS, IMMEDIATE ADOPTION			
Testing Oleoresinous Varnishes (D 154 – 38)	74	0	27
Spec. for Basic Carbonate White Lead (D 81 – 41; ASA K 23 – 1941)	49	0	34
IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Chrome Yellow and Chrome Orange (D 211 – 42 T)	52	0	31
Spec. for Liquid Paint Driers (D 600 – 41 T), as revised	70	0	31
Spec. for C.P. Toluidine Toner (D 656 – 42 T)	52	1	30
Spec. for Dibutylphthalate (D 608 – 41 T)	57	1	43
Testing Liquid Driers (D 564 – 41 T), as revised	71	0	30
Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces (D 610 – 41 T)	52	0	31

The subcommittee is investigating methods for determining conjugation in oils, particularly isomerized oils, and methods for determining the rate of polymerization and acetyl number.

Subcommittee IV on Traffic Paints (Anthony Skett, chairman).—This subcommittee recommends for publication as tentative the Methods of Test for Conducting Road Service Tests, Dry-To-No-Pick-Up-Time, and Light Sensitivity of Traffic Paints.

Work on the establishment of photographic standards for traffic paint failures is under way.

The subcommittee has accumulated considerable data on laboratory tests *versus* road durability tests on traffic paints.

It is also studying the problem of bleeding properties of traffic paints when applied on hard asphalt and tar roads. The subcommittee expects to submit a report on immersion testing of traffic paints in water, dilute caustic, hydrochloric acid, and lime solution.

Subcommittee VII on Accelerated Tests and Protective Coatings (H. A. Nelson, chairman).—This subcommittee recommends for publication as tentative the Method for Evaluating the Degree of Blistering of Organic Coatings When Subjected to Immersion or Other Tests Involving Exposure to Moisture or Liquids.

It also recommends that the Tentative Method for Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron and Steel Surfaces (D 610 – 41 T) be adopted as standard without change.

In view of pending changes, it is recommended that the Tentative Method for Preparation of Steel Panels for Exposure Tests of Enamels for Exterior Service (D 609 – 41 T) be continued as tentative. It is also recommended that the several tentative methods for evaluating exterior paints of the linseed-oil type to chalking (D 659 – 42 T), to checking (D 660 – 42 T), to cracking (D 661 – 42 T), and to erosion (D 662 – 42 T) be continued as tentative.

The subcommittee is preparing methods of test for determining the properties of organic coatings when subjected to immersion and also to humidity.

Considerable progress is reported by the subcommittee in the work of its subgroups.

Group 1 on House Paints (R. W. Matlack, chairman) has given consideration to photographic standards for flaking and scaling and is also studying the question of correlation of accelerated weathering tests with outdoor tint retention tests.

Group 6 on Metal Protective Finishing Systems (J. C. Moore, chairman) has given attention to cleaning methods and the preparation of metal test panels for painting.

Subcommittee VIII on Methods of Analysis of Paint Materials (E. F. Hickson, chairman).—This subcommittee recommends for publication as tentative the following five methods of chemical analysis of extender pigments: Mica Pigment, Diatomaceous Silica Pigment, Barium Sulfate Pigments, Aluminum Silicate Pigment, and Magnesium Silicate Pigment.

Subcommittee IX on Varnish (W. T. Pearce, chairman).—This subcommittee recommends the following revision for immediate adoption in the Standard Methods of Testing Oleoresinous Varnishes (D 154 – 38), and the committee accordingly asks for a nine-tenths vote at the annual meeting in order that this modification may be referred to letter ballot of the Society:

Section 7.—In this section describing the test for nonvolatile matter, add the following note:

> NOTE.—A pyrex or similar heat-resistant glass Petri dish 100 mm. in diameter by 10 mm. in depth may be used in place of the flat-bottom metal dish.

The subcommittee also recommends that the Tentative Specifications for Liquid Paint Driers (D 600 – 41 T) be revised as indicated below and submitted to letter ballot of the Society for adoption as standard:

Section 2.—Add the following as a footnote to the table with a reference to the item for miscibility with oil:

> In case of disagreement between the seller and the purchaser, the test for oil miscibility shall be made at 25 ± 1 C.

The Tentative Methods of Testing Liquid Driers (D 564 – 41 T), revised as follows, are also recommended for adoption as standard:

Section 7.—Add to the description of the miscibility test in this section a note to read as follows:

> NOTE.—In case of disagreement between the seller and the purchaser, the test for oil miscibility shall be made at 25 ± 1 C.

The subcommittee also recommends that the Tentative Method of Test for Phthalic Anhydride Content of Alkyd Resin Solutions (D 563 – 40 T) be revised as follows and continued as tentative:

Section 1.—Add a new footnote 3 to read as follows, placing the footnote reference after the word "content:"

> [3] Other dibasic acids such as maleic also precipitate as potassium salts under the conditions of the test and, if present as esters in the resin would, therefore, be calculated as phthalic anhydride.

The subcommittee is studying methods for measuring the drying time of varnishes and also methods of test for alkyd resin solutions.

Subcommittee XIII on Shellac (W. H. Gardner, chairman).—This subcommittee recommends that the Tentative Method of Test for Color of Orange Shellac (D 29 – 41 T) be continued as tentative due to unsettled conditions of shellac supply. A revision of the Standard Specifications for Orange Shellac (D 237 – 41)[4] is in preparation with the cooperation of Committee D – 9 on Electrical Insulating Materials.

Subcommittee XV on Specifications for Pigments Dry and in Oil When Marketed

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[4] See Editorial Note, p. 258.

in that Form (C. L. Crockett, chairman).—This subcommittee proposed an Emergency Alternate Provision in the Standard Specifications for Mica Pigment (D 607 – 42) to allow an increase in the total residue retained on a No. 325 (44-micron sieve) from "7 per cent" to read "10 per cent." Under existing war conditions the type of scrap mica available does not permit the production of a product conforming to the fineness requirements of Specifications D 607. As mentioned earlier in this report, this proposal was accepted by Committee E-10 and the emergency provisions issued under the designation EA – D 607.

The following revision is recommended in the Standard Specifications for Basic Carbonate White Lead (D 81 – 41) for immediate adoption, and the committee accordingly asks for a nine-tenths affirmative vote at the annual meeting in order that this modification may be referred to letter ballot of the Society:

Section 2 (a).—Change the minimum requirement for lead carbonate from "65.0 per cent" to read "62.0 per cent."

The subcommittee recommends that the Tentative Specifications for Chrome Yellow and Chrome Orange (D 211 – 42 T) and for C.P. Toluidine Toner (D 656 – 42 T) be adopted as standard.

Consideration is being given to revising the Standard Specifications for Mineral Iron Oxide (D 84 – 41).[4]

Subcommittee XVIII on Physical Properties of Materials (M. Rea Paul, chairman).—This subcommittee is preparing a Test for the Preparation of Uniform Paint Films by the Automatic Dip Method. It is also studying the preparation of films for measurement of gloss.

Investigations are being conducted on infra red reflectance and flame testing. The subcommittee is also studying the subject of numerical expression of color differences.

Subcommittee XXV on Cellulose Ester Coatings (R. M. Carter chairman).—This subcommittee recommends that the Tentative Specifications for Dibutylphthalate (D 608 – 41 T) be submitted to letter ballot of the Society for adoption as standard.

Subcommittee XXIX on Painting of Structural Steel and Iron (A. J. Eickhoff, chairman).—This subcommittee is continuing its exposure tests covering an investigation of several methods of surface preparation of steel for painting. Further tests are planned to determine the effect of chemical pretreatment on steel which has been previously painted and then exposed to the atmosphere until rusting occurs.

This report has been submitted to letter ballot of the committee which consists of 231 voting members; 83 members returned their ballots, of whom 61 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. E. SMITH,
Chairman.

M. REA PAUL,
Secretary.
C. H. ROSE,
Assistant Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-1 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:

Red and Brown Iron Oxide Pigments (D 84 – 43 T), superseding the Standard Specifications for Mineral Iron Oxide (D 84 – 41),

Orange Shellac and Other Lacs (D 237 – 43 T), superseding the Standard Specifications for Orange Shellac (D 237 – 41), and

Methyl Ethyl Ketone (D 740 – 43 T).

These recommendations were accepted by Committee E-10 on August 30, 1943, and the new tentative standards appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part II, pp. 117, 135, and 137.

REPORT OF COMMITTEE D-2

ON

PETROLEUM PRODUCTS AND LUBRICANTS

Committee D-2 on Petroleum Products and Lubricants held three regular meetings during the past year (in June at Atlantic City, N. J., in January at Detroit, Mich., and in March at Cleveland, Ohio).

During the year, Subcommittee XXIV on Petroleum Sulfonates, and a new Section IV of Technical Committee C on Turbine Oils, entitled Emulsion Tests, have been organized. The title of Subcommittee XI on Determination of Sulfur, Chlorine and Phosphorus in EP Lubricants has been changed to "Determination of Inorganic Elements in Lubricants." Owing to the broadened scope of the work being done by Section IX on Analysis of Gasoline for Hydrocarbon Types, of Technical Committee A on Gasoline, this section has been discontinued and the work transferred to new Subcommittee XXV on Analysis of Petroleum Products for Hydrocarbon Types.

Table I shows the changes in status during the year of American Standards for petroleum products, approved by the American Standards Association on the recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants.

Subcommittee XIII on Neutralization Number and Saponification (H. P. Ferguson, chairman) has prepared a Proposed Method of Test for Saponification Number of Petroleum Products by Electrometric Titration, appearing in Appendix I, which is presented for publication as information.

Technical Committee C on Turbine Oils (F. C. Linn, chairman) recommends for publication as information the Proposed Method of Test for Oxidation Characteristics of Steam Turbine Oils which appears in Appendix II. Studies carried on in the development of this method by Section III on Oxidation of Turbine Oils (C. Dantsizen, chairman) are described in the report of the section appended hereto.

TABLE I.

Title	A.S.T.M. Designation	ASA Number
New American Standards		
Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue)	D 524 - 42	Z11.47—1942
Test for Tetraethyl Lead in Gasoline	D 526 - 42	Z11.48—1942
Revisions of American Standards		
Test for Gum Content of Gasoline	D 381 - 42	Z11.36—1942
Test for Flash Point by Means of the Pensky-Martens Closed Tester	D 93 - 42	Z11.7 —1942
Test for Melting Point of Paraffin Wax	D 87 - 42	Z11.4 —1942
Test for Vapor Pressure of Petroleum Products (Reid Method)	D 323 - 42	Z11.44—1942

EMERGENCY ALTERNATE PROVISION

During the year, Committee D-2 presented to the Society through Committee E-10 on Standards proposed Emergency Alternate Provisions in the Standard Method of Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524–42). These were accepted by Committee E-10 on July 29, 1942, and issued under the designation EA-D524. They were published in ASTM BULLETIN, No. 118, October, 1942, p. 55, and have been issued in the form of a sticker for attachment to the standard.

I. NEW TENTATIVE STANDARD

The committee recommends that the Method of Test for Oil Content of Paraffin Wax,[1] essentially as published as information in the 1942 report of the committee, be accepted for publication as tentative.

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends revision of six tentative standards as follows:

Tentative Method of Test for Ash Content of Petroleum Oils (**D 482 – 38 T**)[2]:

Section 1.—Add the following note to this section:

> NOTE.—When this method is applied to used oils from engines operating on leaded fuels and to new and used oils containing metallo-organic addition agents, large portions of the metal compounds may be lost by vaporization. In such cases the ash is not a measure either of inorganic contaminating material or of the metallo-organic compounds in the oil. This method is not recommended for use with such oils.

Tentative Method of Test for Aniline Point of Petroleum Products (**D 611 – 41 T**):[2]

The following minor changes are recommended by Subcommittee XVIII on Aniline Point (Eugene Ayres, chairman) to simplify the requirement for the motor and to make it possible to use standard size test tubes manufactured by an extrusion process:

Fig. 1.—Omit the words "variable speed" before the word "motor" in the identification of parts of this figure.

Section 3 (*b*).—Change to read as follows by the addition of the italicized word and figures and the omission of the figures in brackets:

> (*b*) Jacket.—A jacket approximately [38] *37 to 42* mm. in diameter and 175 mm. in length made of heat-resistant glass.

Section 3 (*c*).—Omit the words "variable speed" in the description of the mechanical stirrer.

Tentative Method of Test for Ignition Quality of Diesel Fuels (**D 613 – 41 T**):[2]

A revision of this method involving primarily an editorial rearrangement is recommended by Technical Committee F on Diesel Fuel Oils (C. G. A. Rosen, chairman). The revised method is appended hereto.[3]

Tentative Method of Test for Knock Characteristics of Aviation Fuels (**D 614 – 42 T**):[2]

A revision of this method increasing the minimum allowable air pressure from 26 to 28 in. of mercury based upon tests conducted in the National Bureau of Standards altitude chamber, and other changes primarily of an editorial nature, are recommended by Technical Committee A on Gasoline (C. B. Veal, chairman). The tests at the Bureau showed that the knock rating of some types of fuels varied by nearly one octane unit per inch variation of air pressure. The revised method is appended hereto.[3]

Tentative Method of Test for Knock Characterictics of Motor Fuels (**D 357 – 42 T**):[2]

A revision of this method, largely editorial in nature and including a change in the guide curves in the portion above 90 octane number as a result of cooperative tests made by the Cooperative Fuel Research Committee is recommended by Technical Committee A on Gasoline (C. B. Veal, chairman). The revised method is appended hereto.[3]

Tentative Method of Test for Saponification Number of Petroleum Products

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[1] This method was accepted as tentative by the Society and appears in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 203.

[2] 1942 Book of A.S.T.M. Standards, Part III.

[3] These revised methods were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, pp. 162, 173, and 186, respectively.

by Color-Indicator Titration (**D 94 – 42 T**):[2]

The following minor change is recommended by Subcommittee XIII on Neutralization Number and Saponification (H. P. Ferguson, chairman):

Section 8.—Add a note at the end of the fourth sentence to read as follows:

NOTE.—In the case of insulating oils, the addition of petroleum naphtha is not necessary.

This change was suggested by Committee D-9 on Electrical Insulating Materials which committee has joint jurisdiction with Committee D-2 for this method.

III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in two standard methods of test, as set forth below, and accordingly asks for a nine-tenths affirmative vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Standard Method of Test for Vapor Pressure of Petroleum Products (Reid Method) (**D 323 – 42**):[2]

The following addition is recommended by Subcommittee XXII on Natural Gasoline (G. G. Oberfell, chairman) as a precaution against errors due to presence of carbon dioxide:

Sections 13 and 14.—Add the following note to these sections:

NOTE.—Solid carbon dioxide shall not be used to cool samples in storage or preparatory to the air-saturation step. Carbon dioxide is appreciably soluble in gasoline, and its use has been found to be the cause of erroneous vapor-pressure data.

Standard Viscosity-Temperature Charts for Liquid Petroleum Products (**D 341 – 39**):[2]

The following addition is recommended by Subcommittee V on Viscosity (J. C. Geniesse, chairman) to provide for use of charts C and D at low temperatures:

Section 3.—Add a new paragraph to read as follows:

Kinematic charts C and D may be modified easily for use at low temperatures by substituting the temperatures in column B in the following table for the temperatures in column A which are printed on the bottom of the charts. This modification is based on the mathematical formulas used in the development of the charts. This procedure does not change the scale, therefore the modified charts can be superimposed on the original charts in the overlapping temperature range.

Column A	Column B	Column A	Column B
Temperature Printed on Chart, deg. Fahr.	Low Temperature to be Substituted for those in Column A, deg. Fahr.	Temperature Printed on Chart, deg. Fahr.	Low Temperature to be Substituted for those in Column A, deg. Fahr.
+5	−150	+200	−20
+20	−140	+215	−10
+35	−130	+230	0
+50	−120	+245	+10
+65	−110	+260	+20
+80	−100	+275	+30
+95	−90	+290	+40
+110	−80	+305	+50
+125	−70	+320	+60
+140	−60	+335	+70
+155	−50	+350	+80
+170	−40	+365	+90
+185	−30	+380	+100

IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following two tentative standards be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Method of Test for Carbonizable Substances in Paraffin Wax (**D 612 – 41 T**),[2] on the recommendation of Subcommittee I on Pharmaceutical Tests (V. F. Beyer, chairman), revised as follows to avoid difficulties caused by solidification of the paraffin wax before comparison with the standard is made:

Section 4 (d).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

(*d*) At the end of 10 min. from the time the test tube was first placed in the bath, remove the test tube [and allow to stand in the room for not less than 10 min. nor more than 30 min.], ***immediately*** place [the test tube] ***it*** in the color comparator and compare the acid layer with 5 ml. of the standard colorimetric solution and 5 ml. of white mineral oil in a test tube which has been shaken vigorously for 10 sec. and allowed to stand just long enough for the contents to separate into two layers.

Tentative Method of Test for Carbonizable Substances in White Mineral Oil (*Liquid Petrolatum*) **(D 565 – 41 T)**,[2] on the recommendation of Subcommittee I on Pharmaceutical Tests (V. F. Beyer, chairman).

V. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

The following tentative standards have remained in the tentative status for two years or longer without revision:

Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter (D 155 – 39 T),
Test for Consistency of Lubricating Greases and Petrolatum (D 217 – 38 T),
Test for Sediment in Fuel Oil by Extraction (D 473 – 38 T),
Test for Sulfur in Petroleum Oils by Lamp Method (D 90 – 41 T),
Specifications for Fuel Oils (D 396 – 39 T),
Specifications for Aviation Gasolines (D 615 – 41 T), and
Specifications for Gasoline (D 439 – 40 T).

It is recommended that these tentative standards be continued in the tentative status because further developments are in progress or in prospect.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 73 voting members, with the results shown in Table II.

This report has been submitted to letter ballot of the committee which consists of 73 voting members; 57 members returned their ballots, of whom 56 have voted affirmatively and 0 negatively.

TABLE II.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Test for Oil Content of Paraffin Wax	49	0	8
II. REVISIONS OF TENTATIVE STANDARDS			
Test for Ash Content of Petroleum Oils (D 482 – 38 T)	50	0	6
Test for Aniline Point of Petroleum Products (D 611 – 41 T)	51	0	6
Test for Ignition Quality of Diesel Fuels (D 613 – 41 T)	41	0	16
Test for Knock Characteristics of Aviation Fuels (D 614 – 42 T)	40	0	17
Test for Knock Characteristics of Motor Fuels (D 357 – 42 T)	41	0	16
Test for Saponification Number of Petroleum Products by Color-Indicator Titration (D 94 – 42 T)	51	0	6
III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION			
Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 – 42)	51	0	6
Viscosity-Temperature Charts for Liquid Petroleum Products (D 341 – 39)	56	0	1
IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Test for Carbonizable Substances in Paraffin Wax (D 612 – 41 T), as revised	44	0	13
Test for Carbonizable Substances in White Mineral Oil (Liquid Petrolatum) (D 565 – 41 T)	42	0	15

Respectfully submitted on behalf of the committee,

T. A. BOYD,
Chairman.

R. P. ANDERSON,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-2 submitted to Committee E-10 on Standards a proposed Emergency Method of Test for Color of U. S. Army Motor Fuel (All-Purpose) by Means of an A.S.T.M. Color Standard. This recommendation was accepted by Committee E-10 on August 24, 1943, and the emergency standard appears in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 145, bearing the A.S.T.M. serial designation ES – 32.

REPORT OF SECTION III ON OXIDATION OF TURBINE OILS

TECHNICAL COMMITTEE C ON TURBINE OILS

This section recommends that the Proposed Method of Test for Oxidation Characteristics of Steam-Turbine Oils, as developed by the section, be published as information only as appended hereto.[1]

TABLE I.—RESULTS OF COOPERATIVE OXIDATION TESTS ON SERIES I TURBINE OILS.

Laboratory	Number of Hours to Reach Neutralizaion Number of 2.0			
	Oil No. 1[a]	Oil No. 2[a]	Oil No. 3[b]	Oil No. 4[b]
1	160	200	2030	1020
2	97	77	1740	1140
3	210	75	1700	1420
4[c]	110	50	1275	935
5	87	92	1650	1125
6	140	122	...	1590
7	90	65	1740	1180
8	100	71	2140	1480
Average	124	94	1754	1236
Average Deviation	34	34	190	195
Standard Deviation	40	45	259	218
Max. Deviation from Average	+86	+106	−479	+354

[a] Noninhibited oil.
[b] Oxidation-inhibited oil.
[c] Tests in this laboratory were made at a temperature of 97 C. before the section decided on a temperature of 95 C. which is specified in the proposed test.

Two series of four turbine oils each have been tested by the method with the results shown in Tables I and II. Oil No. 3 of Series I is the same brand as Oil No. 1 of Series II, but the samples were taken from a different shipment.

The average value of the ratio of the average deviations to standard deviations for the eight oils is 0.799. The theoretical value of this ratio is 0.798. When the average deviations are plotted against the average test values, an approximate straight line results, which indicates from the data so far obtained that the average deviation from the average test life is 12 per cent plus 25 hr. Similarly, the data indicate that the standard deviation from the average test life is 15 per cent plus 25 hr.

TABLE II.—RESULTS OF COOPERATIVE OXIDATION TESTS ON SERIES II TURBINE OILS.

Laboratory	Number of Hours to Reach Neutralization Number of 2.0			
	Oil No. 1[b]	Oil No. 2[a]	Oil No. 3[b]	Oil No. 4[a]
1	690	60	2070	60
2	575	85	>1500	80
3	810	120	1450	160
4	1090	75	2310	65
5	835	90	2795	80
6	850	90	1790	60
9	690	75	2200	73
10	750	175	2220	175
Average	786	96	2119	94
Average Deviation	110	26	300	37
Standard Deviation	143	34	391	43
Max. Deviation from Average	+304	+79	+679	+81

[a] Noninhibited oil.
[b] Oxidation-inhibited oil.

Field service data were obtained only on Series II turbine oils. These data are shown in Table III.

Respectfully submitted, on behalf of the section,

C. DANTSIZEN,
Chairman.

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[1] See p. 275.

TABLE III.—FIELD SERVICE DATA ON SERIES II TURBINE OILS.

	Oil No. 1			Oil No. 2		Oil No. 3				Oil No. 4
Make of unit	Westinghouse	Westinghouse	General Electric	General Electric	General Electric	General Electric	General Electric	General Electric	General Electric	Westinghouse
Size of unit, kw	40 000	40 000	2 000	80 000	80 000	2 000	30 000	30 000	30 000	72 000+
Capacity of oil system, gal	1 870	2 000	140	3 600	2 500	140	900	900	900	3 800
Amount of makeup added, gal	1 664	2 150	245	10 856	10 856	376	1 202	591	378	3 150
Number of service hours	51 666	48 699	19 723	73 487	64 204	15 537	36 948	25 397	25 466	16 000
Normal bearing-oil discharge temperature deg. Fahr	130 to 140	130 to 140	150 to 175	117 to 145	113 to 140	130 to 170	130 to 140	130 to 140	130 to 140	125 to 140
Analysis of oil:[a]										
Neutralization number	0.06	0.03	0.01	1.59	1.70	0.06	0.06	0.06	0.06	1.80
Steam emulsion number	290	210	225	311	331	105	275	137	227	...
Saybolt Universal viscosity at 100 F., sec	155	152	...	167	167	...	...	...	...	182
Sludge	none	none	none	cloudy	cloudy	none	none	none	none	heavy
Makeup added per year, gal	282	386	124	1 295	1 480	212	286	204	130	1 725
Percentage annual make up	15	19	89	36	59	152	32	23	14	45

[a] The analysis of Series II turbine oils when new is as follows:

	Oil No. 1	Oil No. 2	Oil No. 3	Oil No. 4
Neutralization number	0.01	0.02	0.01	0.01
Steam emulsion number	20	40	20	...
Saybolt Universal Viscosity at 100 F., sec	149	150	150	150
Sludge	none	none	none	none

APPENDIX I

PROPOSED METHOD OF TEST FOR SAPONIFICATION NUMBER OF PETROLEUM PRODUCTS BY ELECTROMETRIC TITRATION[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test is designed to indicate in oxidized petroleum products the presence and concentration of constituents having acidic or basic characteristics after saponification with an excess of strong base, and to resolve these constituents into groups having weak-acid, strong-acid, weak-base, and strong-base ionization properties. It is intended for the determination of saponification number of new and used motor oils, electrical oils, and greases whether compounded or not. It may be used for the identification of unmixed animal or vegetable oils, and for the determination of the amount of fatty material in compounded products, provided the sample does not contain appreciable amounts of nonfatty compounds that consume alkali or acid under the conditions of the test such as certain compounds of sulfur, phosphorus, the halogens, nitrogen, and metals other than potassium and sodium. This method is not applicable to samples containing appreciable amounts of a strong base.

Note 1.—A color-indicator titration method is also available in the Tentative Method of Test for Saponification Number of Petroleum Products by Color-Indicator Titration (A.S.T.M. Designation: D 94) of the American Society for Testing Materials.[2] The two methods produce comparable results when they are applied to new or slightly colored oils or to animal and vegetable oils. The electrometric method is to be preferred, and is considered to be more reliable in those cases in which the oil has a high inherent color or forms stable emulsions with the ethanol saponification medium used in the color-indicator method. No general relationship is known between the degree of oxidation of the oil and the saponification number as determined by either of these two methods.

Note 2.—This method is applicable to materials that are soluble, or nearly soluble, in benzene-isopropylalcohol mixtures. It is equally applicable to materials that are colorless or colored or that produce colored solutions during the saponification treatment. Unchanged compounds that are only weakly acidic or basic, and whose dissociation constants, K_a or K_b, in water are equal to or less than $K = 10^{-9}$, are not detectable and do not interfere; salts react if their hydrolysis constants are greater than $K = 10^{-9}$. Various groups, such as esters of strong and weak acids, that upon saponification produce acids with different degrees of ionization may be distinguished provided the dissociation constant of the more highly ionized acid group is at least 1000 times that of the weaker acidic group. Elementary sulfur and compounds of sulfur, phosphorus, the halogens, nitrogen, and metals other than potassium or sodium may increase the saponification number by reacting with the potassium hydroxide to form acids or insoluble compounds.

Definitions

2. (*a*) *Saponification Number.*—The saponification number is the number of

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1943.

[2] 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 207.

milligrams of potassium hydroxide required to react with the acidic constituents present after saponifying one gram of sample.

NOTE 3.—The saponification number expresses the total amount of acid or base-consuming constituents that were initially present or formed during the saponification, and includes the constituents with strong-acid characteristics that react under the conditions of the test as prescribed in this method.

(*b*) *Strong-Acid Saponification Number.*—The strong-acid saponification number is the number of milligrams of potassium hydroxide required to react with the constituents with strong-acid characteristics present after saponifying one gram of sample.

NOTE 4.—The difference between the saponification number and the strong-acid saponification number expresses the amount of weak-acid constituents present or formed during the saponification.

(*c*) *Base Saponification Number.*—The base saponification number is the number of milligrams of potassium hydroxide equivalent to the amount of acid required to neutralize the basic constituents present after saponifying one gram of sample.

NOTE 5.—Base saponification number expresses the total amount of constituents having basic characteristics.

Outline of Test

3. The sample, dissolved in a medium of equal parts of benzene and an isopropyl alcohol solution of potassium hydroxide, is refluxed for 2 hr. after which the excess potassium hydroxide is titrated with alcoholic hydrochloric acid, using as indicator the potential between a glass electrode and a calomel electrode. In most cases, the curve of potential against volume of titrating solution shows an inflection point at the end point; if not, a defined potential is taken as the end point.

Apparatus

4. The following apparatus described in the Appendix shall be used: meter, glass electrode, reference electrode, stirrer, special 50-ml. burette, special 250-ml. tall-form beaker, titration stand, reflux condenser, optional saponification vessel, and hot plate.

Reagents

5. (*a*) *Standard Potassium Hydroxide* (*0.2 N in anhydrous isopropyl alcohol*).—Add 12 to 13 g. of KOH (A.C.S. analytical reagent grade) to approximately 1 liter of c.p. anhydrous isopropyl alcohol. Boil the mixture gently under reflux for 10 min. Add approximately 3 g. of c.p. barium hydroxide[3] and boil gently under reflux for 10 min. Cool to room temperature and filter the supernatant liquid through a fine glass filtering funnel. Store the solution in a chemically resistant bottle, and dispense it in a manner such that it is protected from atmospheric carbon dioxide by means of a guard tube containing soda lime or ascarite and does not come in contact with cork or rubber.

(*b*) *Standard Hydrochloric Acid* (*0.2 N in isopropyl alcohol*).—Add 18 to 19 ml. of c.p. HCl (sp.gr. 1.19) to 1 liter of c.p. anhydrous isopropyl alcohol. Standardize the solution frequently enough to detect changes of 0.0005 *N* against a standard base or pure sodium carbonate. It is preferable to standardize by potentiometric titration against 0.325 g. of analytical grade c.p. anhydrous sodium carbonate diluted with 190 to 200 ml. of distilled water.

(*c*) *Isopropyl Alcohol.*—C.P. anhydrous isopropyl alcohol containing less than 0.1 per cent water by test.

(*d*) *Benzene.*—C.P. anhydrous benzene (C_6H_6).

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[3] Barium hydroxide is insoluble in anhydrous isopropyl alcohol, and any excess above that required to react with the carbonate is removed along with the barium carbonate in the subsequent filtration.

(*e*) *Standard Aqueous Buffer Solution, pH = 4 (for calibrating the glass electrode).*—Dissolve 5.105 g. of analytical grade potassium acid phthalate in CO_2-free distilled water and dilute to exactly 500 ml. This solution must be made up fresh when needed. The pH of this buffer solution is taken as 4.00 at 25 C. and is accepted as being constant at all temperatures between 15 and 35 C. for the purposes of this method. The correct glass electrode potential, *cG*, of this solution shall be taken as 0.236 v. at 25 C.

(*f*) *Standard Nonaqueous Buffer Solution, cG = 0.650 v. (for calibrating the glass electrode).*—Prepare a 0.1 *M* solution of pure thiophenol in c.p. isopropyl alcohol. Add 10 ml. of the 0.1 *M* thiophenol solution to 100 ml. of titration solvent, and add an amount of standard 0.2 *N* KOH solution (Paragraph (*a*)) equivalent to 7.50 ml. of 0.1 *N* KOH solution. Mix thoroughly and use immediately.

(*g*) *Standard Nonaqueous Buffer Solution, cG = 0.236 V., (for calibrating the glass electrode).*—Prepare a 0.1 *M* solution of pure trichloroacetic acid in c.p. isopropyl alcohol. Add 10 ml. of the 0.1 *M* trichloroacetic acid solution to 100 ml. of titration solvent, and add an amount of standard 0.2 *N* KOH (Paragraph (*a*)) equivalent to 5.25 ml. of 0.1 *N* KOH solution. Mix thoroughly and use immediately.

(*h*) *Potassium Chloride Electrolyte.*—Prepare a saturated solution of c.p. KCl in water.

(*i*) *Benzene-Isopropylalcohol Wash Solution.*—Mix 500 ml. of c.p. benzene with 500 ml. of c.p. anhydrous isopropyl alcohol.

Preparation of Apparatus

6. (*a*) *Electrodes, Preparation for Use and Maintenance.*—The electrode system requires regular attention to obtain constant and reproducible values. Before and after using, wipe the glass electrode thoroughly with a piece of clean cloth, and rinse with distilled water. Also, wipe the calomel electrode with a piece of clean cloth, carefully remove the ground-glass sleeve, and thoroughly clean each ground surface by wiping with the cloth. Flush the ground-glass contact joint (salt bridge) by allowing a few drops of the potassium chloride electrolyte to drain through. Wet the ground surfaces thoroughly with potassium chloride electrolyte, and replace the glass sleeve. With the sleeve firmly in place to eliminate possible leakage, rinse the calomel electrode well with distilled water.[4] Prior to each titration, soak the prepared electrodes for at least 2 min. in distilled water. When not in use, keep the lower half of each electrode immersed in distilled water. Also, frequently clean the glass electrode by immersion in cold chromic acid solution such as is used for cleaning glass vessels. Once each week drain all the electrolyte from the calomel cell and replace with fresh potassium chloride electrolyte.

The calomel electrode shall contain, at all times, sufficient saturated potassium chloride electrolyte so that the level of the salt solution inside the calomel electrode is above the level of the liquid in the titration beaker. Although the electrodes are not extremely fragile, they should be handled carefully at all times.

(*b*) *Calibration of Electrode System and Calculation of cG.*—To calibrate the electrodes after preparation, place sufficient standard buffer solution in the beaker to cover the lower half of the electrodes and stir for several minutes while maintaining the temperature of the buffer solution within plus or minus 2.0 C. of the temperature at which the titrations are to be made. Measure the potential, E, in volts, between the electrodes. The cell constant, E_{GC}[5] for the titration cell shall be calculated from the expression:

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[4] Keeping the ground-glass joint free of foreign material is very important in obtaining reproducible potentials in the titration cell, since contamination may introduce uncertain and unnoticeable liquid contact potentials. While this is of secondary importance when titration end points are chosen by inflection points, it may be quite serious when titration end points are taken at fixed cell potentials.

$$E_{GC} = E - 0.236$$

Then, the correct glass electrode potential, cG, in any subsequent titration made at a temperature within plus or minus 2.0 C. of the calibration temperature, will be the difference between the observed potential, E, at that point and the cell constant, or

$$cG = E - E_{GC}$$

The E_{GC} value of most glass-electrode-calomel-electrode systems will have a value approximately equal to -0.450 v. In the mathematical relations given, E bears the sign indicated by a voltmeter with the glass electrode connected to the negative pole of the meter (the negative pole is the pole to which the zinc terminal of a common dry cell must be connected to give an up-scale reading on the meter).

In the event that the meter is connected to operate as a pH meter, and the pH scale on the meter is being read, vary the so-called "asymmetry potential" adjustment until the meter registers a scale reading, in pH units, equal to 4.00 while the electrodes are immersed in the standard aqueous buffer solution. In this case, the correct glass electrode potential, cG, in any subsequent titration made within plus or minus 2.0 C. of the calibration temperature, T, will be directly proportional to the meter reading in pH units and shall be calculated as follows:

$$cG = 0.000198\ T \times \text{pH meter reading}$$

where T = calibration temperature in degrees Kelvin.

(*c*) *Performance Test of Electrode System and Meter.*—When dipped into a mixture of 50 ml. of benzene and 50 ml. of isopropyl alcohol containing 0.5 to 0.75 ml. of 0.2 *N* alcoholic KOH in excess, the potential between the glass and reference electrodes shall have changed more than 0.480 v. (8 pH units) from the potential between the same electrodes dipped in the standard aqueous buffer solution (pH = 4, Section 5 (*e*)).

(*d*) *Calibration of cG = 0.236 v. and cG = 0.650 v. Reading of Electrode System.*—To calibrate the electrode system at cG = 0.236 v. and cG = 0.650 v. with the standard nonaqueous buffer solutions, prepare the electrodes carefully as described in Paragraph (*a*), and immerse them consecutively in the respective nonaqueous buffer solutions (Section 5 (*f*) and (*g*)). Stir for 5 min., maintaining the temperature of each buffer solution within plus or minus 2 C. of the temperatures at which the titrations are to be made. Read the potential between the electrodes, calculate the apparent cG of the solutions as described in Paragraph (*b*), and record this value as the standard cG = 0.236 v. and cG = 0.650 v. value of the electrode system for selecting end points.

Procedure

7. (*a*) *Size of Sample.*—The size of sample shall be as follows:

Saponification Number	Weight of Sample, g.
0.0 to 5.0	20.0 ± 0.05
5.0 to 30.0	5.0 ± 0.02
30.0 to 225	1.0 ± 0.005
Over 225	0.5 ± 0.002

(*b*) *General Procedure.*—The following procedure is intended for samples of unknown saponification characteristics or for samples that upon saponification produce a precipitate that clings to the surface of the reflux vessel:

Weigh the sample into a 250-ml. tall-form, lipless beaker and add 40 to 45 ml. of c.p. benzene. From the 50-ml. burette, add 40.0 ± 0.05 ml. of 0.2 *N* alcoholic KOH allowing 60 to 80 sec. to elapse between adding the first 39.5 ml. and the final 0.5 ml. Place the reflux condenser in the beaker, cool the condenser by passing tap water at a temperature less than 35 C. through it, and heat on a hot plate at refluxing temperature for 110 to 120 min. after the refluxing begins (Note 6).

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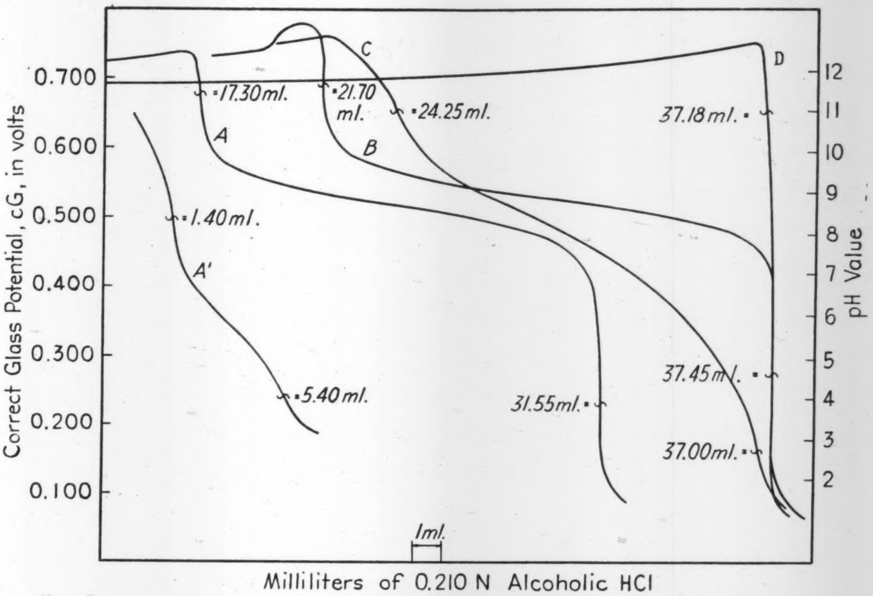
[5] E_{GC} is a constant only in the sense that it combines all voltage effects that should not vary throughout a titration due to a variation in the hydrogen ion activity. For this reason, it must be determined as described at the temperature that is to be used, and redetermined at intervals thereafter.



A	1.00 g. of blown rapeseed oil, solvent curve
A′	1.00 g. of blown rapeseed oil, water curve
B	1.00 g. of castor oil, direct titration curve
C	10.00 g. of black oxidized motor oil, direct titration curve
D	Blank, direct titration curve

FIG. 1.—Illustrative Titration Curves.

Disconnect the condenser and wash the exposed condenser and beaker surfaces with 10 to 20 ml. of benzene-isopropylalcohol wash solution. Cool the beaker and contents to room temperature and transfer the contents of the saponification beaker to a 250-ml., tall-form, lipless beaker, rinse the saponification beaker three times with 5-ml. portions of benzene-isopropylalcohol wash solution and cover with a watch glass. Add 100 ml. of CO_2-free distilled water to the saponification beaker and heat just to boiling. Cool the beaker and contents to room temperature and cover with a watch glass.

Titrate the solutions as described below within 2 hr. after the end of the saponification period (Note 7). For highly accurate results, purge the space above the liquid in the reflux vessel and in the beakers with a stream of CO_2-free air.

Prepare the calomel and glass electrodes as directed in Section 6, and connect the frame of the motor and meter to a suitable ground to prevent electrostatic interference. Place the beaker, containing the benzene-isopropylalcohol solution, on the titration stand, adjust so that the lower half of each electrode is immersed in the titration solution, start the mechanical stirrer and adjust the speed of stirring so that there is vigorous stirring without spattering. Fill the 50-ml. burette with 0.2 *N* alcoholic HCl, and place the burette in position in the titration assembly so that the tip is immersed approximately 1 in. in the titration solution. Record the initial reading of potential between the electrodes. Add the 0.2 *N* alcoholic HCl at a slow steady rate, approximately 1 ml. per min., until the potential between the electrodes levels off and tends to decrease. (Stop the addition of 0.2 *N* alcoholic HCl immediately when the decrease of potential is definite and noticeable in order to avoid overtitrating the first end point, see Fig. 1.) Close the stopcock of the burette, wait until an unchanging potential[6] has been established and record the meter and burette readings. Add further small portions (as stated below) of 0.2 *N* alcoholic HCl and, after waiting until an unchanging potential[6] has been established, record the meter and burette readings in each case. When the *cG* potential is above 0.513 v. (corresponding to pH scale reading of 8.8), add 0.10-ml. portions. In the region where the *cG* potential lies between 0.513 and 0.432 v. (corresponding to pH scale reading of 8.8 and 7.3, respectively), add 0.50-ml. portions. When the *cG* potential is below 0.432 v. (corresponding to pH scale reading of 7.3) add 0.10-ml. portions. Titrate in this manner until the potential of the cell changes less than 0.005 v. (corresponding to 0.1 pH units) per 0.10 ml. when the potential between the electrodes indicates a *cG* potential less than 0.118 v. (corresponding to pH scale reading of 2.0). Remove the titrated solution, wash the electrodes with isopropyl alcohol and immerse the electrodes in distilled water.

Prepare the calomel and glass electrodes as before. Place the beaker containing the water solution of the insoluble residue on the titration stand, adjust so that the lower half of each electrode is immersed in the solution, start the mechanical stirrer and adjust the speed of stirring so that there is vigorous stirring without spattering. Fill the 50-ml. burette with 0.2 *N* alcoholic HCl, and place the burette in position in the titration assembly so that the tip is immersed approximately 1 in. in the solution. Record the initial reading of potential between the electrodes. Add the 0.2 *N* alcoholic HCl at a steady rate until a *cG* potential of 0.532 v. (corresponding to pH scale reading of 9.0) is reached. Close the stopcock of the burette, wait until the potential does not change noticeably[7] and record the meter and burette reading. Add suitable small portions (as stated below) of 0.2 *N* alcoholic HCl and, after waiting until the potential does not change noticeably,[7] record the meter and burette reading. Add 0.10-ml. increments as long as more than 0.03-v. (corresponding to 0.5 pH units) changes in potential result. Otherwise, increase the increments to a size that produces a 0.03-v. (corresponding to 0.5 pH units) change in potential. Titrate in this manner until the potential of the cell changes less than 0.005 v. (corresponding to 0.1 pH units) per 0.10 ml. when the potential between the electrodes indicates a *cG* potential less than 0.118 v. (corresponding to pH scale reading of 2.0). Remove the titrated solution, wash the electrodes with water and immerse the electrodes in distilled water.

Plot a graph using milliliters of 0.2 *N* alcoholic HCl as abscissas and millivolts (or pH scale readings) as ordinates. Typical curves are shown in Fig. 1. On the curve from the benzene-isopropyl alcohol titration, mark inflection points that occur in the neighborhood of *cG* potentials of 0.65 and 0.24 v. (corresponding to pH scale readings of 10.5 and 4.0, respectively). On the curve from the water solution titration, mark inflection points that occur near *cG* potentials of 0.49 and 0.30 v. (corresponding to pH scale reading 8.2 and 5.0, respectively).

---

[6] Consider the cell potential constant when it changes less than 0.005 v. (corresponding to 0.1 pH unit) per min. This may require from 1 to 2 min. per 0.100 v. (corresponding to 1.7 pH units) change in potential when adding 0.05-ml. increments.

[7] Generally, this state is reached in 15 to 30 sec.

NOTE 6.—If the optional saponification vessel is used (see Appendix, Paragraph (*i*)), do not use grease on the ground-glass joint but moisten it with isopropyl alcohol to prevent seizure of the joint.

NOTE 7.—Whenever the water solution has little or no color, the titration of the water solution may be carried out colorimetrically using phenolphthalein and methyl orange indicators to indicate end points at pH of 8.2 and 5.0, respectively.

(*c*) *Direct Titration Procedure.*—The following procedure may be used for samples of known saponification characteristics and for samples that upon saponification do not produce a precipitate that clings to the surface of the reflux vessel.

The determination shall be made as directed in Paragraph (*b*), except that a single titration shall be made directly in the saponification beaker without transfer of the saponified solution to another beaker. The details of the saponification titration and selection of end points shall be the same as specified in Paragraph (*b*) except that the preparation and titration of the water solution shall be omitted.

(*d*) *Modified Procedure.*—In those cases in which the titration curve or curves of the saponified sample have no definite inflection points the determination shall be made as directed in Paragraph (*b*) or (*c*) except that the end points shall be marked at *cG* potential of 0.650 and 0.236 v. (corresponding to pH scale reading of 11.0 and 4.0, respectively) for the titration curves of the benzene-isopropylalcohol solution and at *cG* potential of 0.485 and 0.295 v. (corresponding to pH scale reading of 8.2 and 5.0, respectively) for the titration curves of the water solution. The details of the saponification and titration shall be the same as specified in Paragraph (*b*) or (*c*).

NOTE 8.—In case the *cG* potential of 0.650 v. falls in the horizontal portion of the curve denoting an excess of strong base, or falls above the curve entirely, or falls in the transitory curved portion of the graph where the slope of the curve begins to dip away from the horizontal, then the arbitrary end point shall be chosen 0.025 v. (corresponding to 0.4 pH units) below the point at which the transitory curved portion enters a region of greatest or constant slope.

The emf. between the electrodes corresponding to $cG = 0.236$ v. and 0.650 v. as calculated in Section 6 (*b*), may be used as the correct value for routine determinations with a given electrode system. For work of a special nature or for cooperative and referee testing, the potentials corresponding to $cG = 0.236$ v. and $cG = 0.650$ v. should be determined according to Section 6 (*d*).

(*e*) *Procedure for Blank Determinations.*—Simultaneously with the sample determinations, make duplicate blank determinations exactly following the procedure used for the sample determination except for omission of the sample. Blank determinations shall be made on each batch of alcoholic KOH solution, alcoholic HCl solution, benzene, and isopropyl alcohol. Mark inflection points[8] and end points on the blank titration curves as directed in the procedure used.

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[8] When the solvents used are very pure, the blank non-aqueous or aqueous titration curves may exhibit a straight line break and may not show two inflection points. When this condition is approached mark end points as indicated in Section 7(*d*).

Calculations and Report

8. (*a*) Calculate the saponification, strong-acid saponification, and base saponification numbers as follows:

Saponification number

$$= \frac{56[(A_b + a_b) - (A_s + a_s)] \times N}{W}$$

Strong-acid saponification number

$$= \frac{56[(B_b + b_b) - (B_s + b_s)] \times N}{W}$$

$= 0.0$ when $(B_b + b_b)$ is equal to or less than $(B_s + b_s)$

Base saponification number

$$= \frac{56[(B_s + b_s) - (B_b + b_b)] \times N}{W}$$

$= 0.0$ when $(B_s + b_s)$ is equal to or less than $(B_b + b_b)$

where:

A_s = milliliters of standard HCl solution required to neutralize the excess base in the titration of the benzene-isopropylalcohol solution down to the end point at which the *cG* potential is approximately equal to 0.65 v. (corresponding to pH scale reading of 11.0),

A_b = corresponding value for the blank titration,

a_s = milliliters of standard HCl solution required to neutralize the excess base in the titration of the water solution down to the end point at which the *cG* potential is approximately equal to 0.485 v. (corresponding to pH scale reading of 8.2),

a_b = corresponding value for the blank titration,

B_s = milliliters of standard HCl solution required to neutralize the basic materials in the titration of the benzene-isopropylalcohol solution down to the end point at which the *cG* potential is approximately equal to 0.236 v. (corresponding to pH scale reading of 4.0),

B_b = corresponding value for the blank titration,

b_s = milliliters of standard HCl solution required to neutralize the basic materials in the titration of the water solution down to the end point at which the *cG* potential is approximately equal to 0.295 v. (corresponding to pH scale reading of 5.0),

b_b = corresponding value for the blank titration,

N = normality of the standard HCl solution, and

W = weight of the sample in grams.

(*b*) The calculated figures shall be reported as saponification number, "strong-acid saponification number," or "base saponification number."

Reproducibility of Results

9. Results obtained in different laboratories should not differ from the mean by more than the following amounts:

Type of Titration Curve	Permissible Difference, ml. of 0.2 *N* acid
Good inflection point	0.10
No inflection point	0.20

APPENDIX

APPARATUS

The apparatus shall consist of the following:

(a) *Meter.*—A voltmeter or potentiometer that will operate with an accuracy of plus or minus 0.005 v. and a sensitivity of plus or minus 0.002 v. over the entire range of plus or minus 1.0 v., and will operate on an input of less than 5×10^{-12} amp., when an electrode system having 1000 megohms resistance is connected across the meter terminals. The meter shall be enclosed in a metal shield connected to the ground; a satisfactory terminal shall be provided to connect the shielded connection wire from the glass electrode to the meter without external exposure at any point to electrostatic fields. A continuous reading electronic voltmeter is satisfactory and desirable.

(b) *Glass Electrode.*—A pencil-type glass electrode (*C*, Fig. 2) 12.5 to 18.0 cm. (4.9 to 7.1 in.) in length and 0.8 to 1.4 cm. (0.31 to 0.55 in.) in diameter. The body of the electrode shall be made of a chemically resistant glass tube with a wall thickness of 0.1 to 0.3 cm. (0.039 to 0.117 in.). The end dipping into the solution shall be closed with a hemisphere of Corning 015 glass sealed onto the electrode tube, and the radius of this hemisphere shall be about 0.7 cm. (0.28 in.). The thickness of the glass in the hemisphere shall be great enough so that the resistance of the hemisphere is 200 to 1000 megohms. The electrode shall contain a reproducible, permanently sealed liquid cell for making electrical connection with the inner surface of the hemisphere. The entire electrical connection from the sealed contact cell to the meter terminal shall be surrounded by an electrical shield that will prevent electrostatic interference when the shield is grounded. The shield shall be insulated from the electrical connection by insulating material of the highest quality, such as rubber and glass, so that the resistance between the shield and the electrical connection is greater than 50,000 megohms.

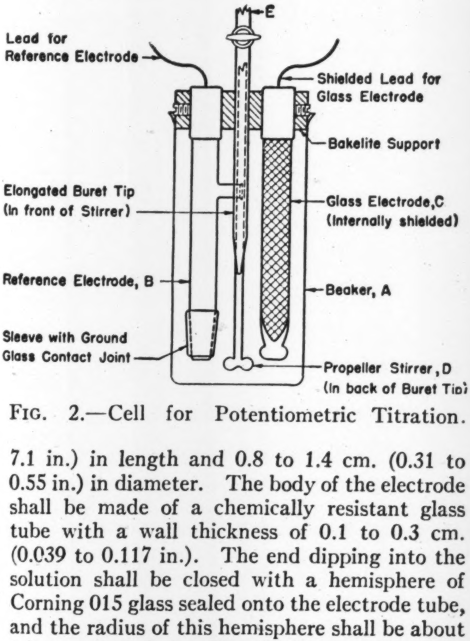
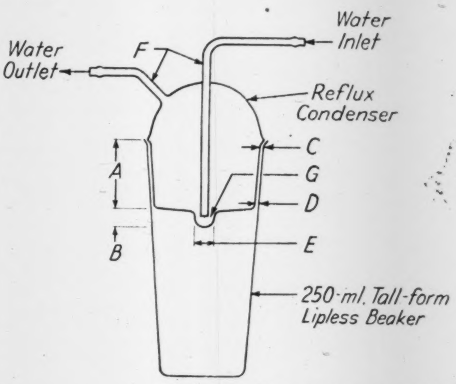

E
Lead for Reference Electrode
Shielded Lead for Glass Electrode
Bakelite Support
Elongated Buret Tip (In front of Stirrer)
Glass Electrode,C (Internally shielded)
Reference Electrode, B
Beaker, A
Sleeve with Ground Glass Contact Joint
Propeller Stirrer, D (In back of Buret Tip)

FIG. 2.—Cell for Potentiometric Titration.

(c) *Reference Electrode.*—A pencil-type calomel electrode (*B*, Fig. 2) 12.5 to 18.0 cm. (4.9 to 7.1 in.) in length and 0.8 to 1.4 cm. (0.31 to 0.55 in.) in diameter. This electrode shall be made of glass and shall be provided with an external, removable glass sleeve on the sealed end that is dipped into the titration solution. The glass sleeve shall be 0.8 to 2.5 cm. (0.31 to 0.99 in.) in length, shall be slightly tapered, and shall be ground to fit the electrode so that the sealed end of the electrode protrudes 0.2 to 2.0 cm. (0.08 to 0.79 in.) beyond the sleeve. The ground surfaces shall be continuous and free of smooth spots. At a point midway between the extremities of the ground surface, the electrode tube shall be pierced by a hole or holes 0.1 cm. (0.039 in.) in diameter. The electrode shall contain the necessary mercury, calomel, and electrical connection to the mercury, all arranged in a permanent manner. The elec-


Water Inlet
Water Outlet
F
Reflux Condenser
A
B
C
G
D
E
250-ml. Tall-form Lipless Beaker

A	40 to 50 mm.	(1.5 to 2.0 in.)
B	5 to 10 mm.	(0.2 to 0.4 in.)
C (Clearance)	0.5 to 3.5 mm.	(0.02 to 0.14 in.)
D (Clearance)	0.5 to 2.0 mm.	(0.02 to 0.08 in.)
E (Diameter)	10 to 16 mm.	(0.39 to 0.62 in.)
F (Inside Diameter)	4 to 7 mm.	(0.16 to 0.27 in.)
G (Clearance)	2 to 4 mm.	(0.08 to 0.16 in.)

FIG. 3.—Saponification Reflux Vessel.

trode shall be filled almost to capacity with saturated potassium chloride electrolyte (Section 4 (*h*)) and shall be equipped with a stoppered port through which the electrolyte may be replenished. When suspended in the air and with the sleeve in place, the electrode shall not leak electrolyte at a rate greater than 1 drop in 10 min.

(*d*) *Stirrer.*—A variable-speed mechanical stirrer, of any suitable type, equipped with a glass, propeller-type stirring paddle (*D*, Fig. 2). A propeller with blades 1 cm. (0.39 in.) in radius and set at a pitch of 30 to 45 deg. is satisfactory. The stirring apparatus must be electrically correct and grounded so that connecting or disconnecting the power to the motor will not produce an appreciable permanent change in meter reading during the course of the titration.

(*e*) *Burette.*—A burette of 50-ml. capacity, graduated in 0.10-ml. divisions, calibrated with an accuracy of plus or minus 0.05 ml., and provided with a tip that extends 10 to 13 cm. (4 to 5 in.) beyond a glass stopcock.

(*f*) *Beaker.*—A tall-form, lipless glass beaker (*A*, Fig. 2) of 250-ml. capacity, made of chemically resistant glass. The beaker shall be 12 to 13 cm. (4.7 to 5.1 in.) in length, and shall be 5.5 to 6.5 cm. (2.15 to 2.55 in.) in diameter at the open end.

(*g*) *Titration Stand.*—A suitable titration stand to support the electrodes, stirrer, and burette in the position shown in Fig. 2. An arrangement that allows the removal of the beaker without disturbing the electrodes, burette, and stirring paddle is satisfactory.

(*h*) *Reflux Condenser.*—A suitable immersion-type condenser, made to fit loosely the top of the tall-form, lipless 250-ml. beaker, and made of chemically resistant glass.[9] It shall conform to the dimensions shown in Fig. 3 except that the manner of attaching the water inlet and outlet ports may be modified as desired.

(*i*) *Optional Saponification Reflux Vessel.*—Optional only for saponifications that necessitate transfer of the saponified sample to another vessel before titration. An Erlenmeyer flask, 300-ml. capacity, made of chemically resistant glass[9] to which an adequate Allihn-type reflux condenser is attached by means of a ground-glass water-cooled joint.

(*j*) *Hot Plate.*—A suitable hot plate heated by electricity or steam.

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[9] Pyrex and Kimble chemically resistant glasses are suitable.

APPENDIX II

PROPOSED METHOD OF TEST FOR OXIDATION CHARACTERISTICS OF STEAM-TURBINE OILS[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test is intended for the determination of the oxidation characteristics of steam-turbine oils including those used for steam-turbine gears.

Outline of Method

2. The oil sample is subjected to a temperature of 95 C. in the presence of water, oxygen, and an iron-copper catalyst, and the time required to build up a neutralization number of 2 in the sample is determined.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Oxidation Cell.*—An oxidation cell as shown in Fig. 1. The component parts of the cell comprise a test tube, a mushroom condenser, and an oxygen delivery tube with a fritted-glass outlet, all made of pyrex glass and with dimensions as shown in Fig. 1.

(*b*) *Oil Bath.*—A thermostatically controlled oil bath capable of maintaining a temperature of 95 ± 0.5 C. (203 ± 0.9 F.) in the oil sample in the oxidation cell, fitted with a suitable stirring device to provide a uniform temperature throughout the bath and large enough to hold the desired number of oxidation cells immersed in oil to a depth of approximately 350 mm.

(*c*) *Flow Meter.*—A flow meter with a capacity of at least 3 l. per hr. calibrated with an accuracy of plus or minus 0.1 liter per hr.

(*d*) *Oxygen Supply.*—Oxygen supply with suitable pressure-regulating mechanism.

(*e*) *Catalyst.*—Open-hearth iron wire[2], 0.064 in. (1.63 mm.) in diameter, No. 14 A.w.g.; and electrolytic copper wire, 0.064 in. (1.63 mm.) in diameter, No. 14 A.w.g.

Preparation of Catalyst Coils

4. The catalyst coils shall be prepared on the day they are to be used in accordance with the procedure described in the following Paragraphs (*a*) and (*b*).

(*a*) Clean a 3-m. length of the iron wire and an equal length of the copper wire with wads of absorbent cotton wet with A.S.T.M. precipitation naphtha[3] followed by abrasion with abrasive paper[4] until a fresh metal surface is exposed. Then wipe with dry absorbent cotton until all loose particles of metal and abrasive have been removed (Note 1).

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1943.

[2] Armco iron wire or equivalent is satisfactory.

[3] Requirements for this precipitation naphtha are given in Section 4 of the Standard Method of Test for Precipitation Number of Lubricating Oils (A.S.T.M. Designation: D 91) of the American Society for Testing Materials, see 1942 Book of A.S.T.M. Standards, Part III, p. 196.

[4] No. 00 Garnet paper carborundum brand or equivalent is satisfactory.

NOTE 1.—After the catalyst wires have been cleaned, they should be handled in subsequent operations with wads of dry absorbent cotton or cotton gloves to prevent the wires from coming in contact with the skin.

(*b*) Twist the iron and copper wires tightly together at one end for approximately six turns and then wind them simultaneously alongside each other on a mandrel, the dimensions of which are given in Fig. 2, inserting the iron wire in the deeper thread. Twist the free ends of the iron and copper wires together for approximately six turns, remove from the mandrel and, if necessary, increase the length of the coils so that when they are placed over the oxygen delivery tube with one end resting on the fritted-glass outlet, the other end will reach to the upper surface of the oil sample in the cell after the water is added.

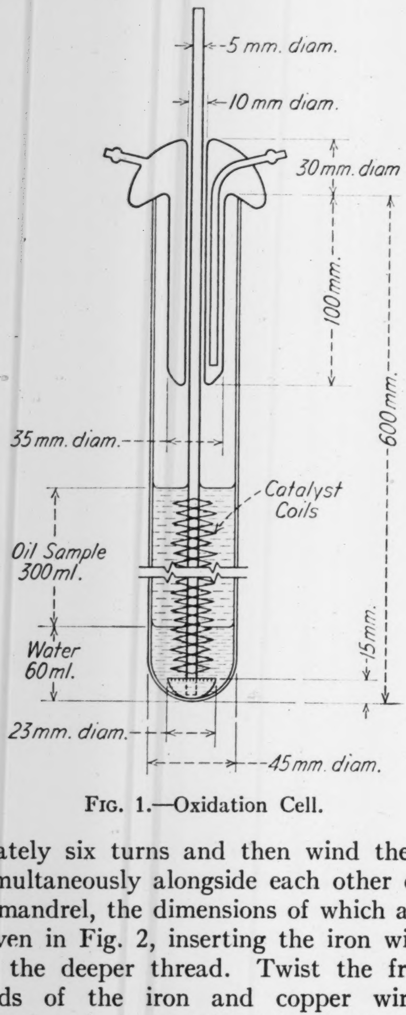

FIG. 1.—Oxidation Cell.

Preparation of Oxidation Cell

5. (*a*) *New Oxidation Cell.*—Clean new test tubes, oxygen delivery tubes, and condensers by rinsing each consecutively with acetone, tap water, chromic acid cleaning solution, and then with tap water until all acid is removed. Rinse twice with small portions of acetone and three times with distilled water. Finally, fill the test tubes with distilled water, insert the oxygen delivery tubes and condensers and store in this condition until ready to use or at least 24 hr. Just prior to use, empty and drain the test tubes and dry the outside of the oxygen delivery tubes and condensers with absorbent cotton.

(*b*) *Used Test Tubes.*—Clean used test tubes by rinsing and scrubbing with A.S.T.M. precipitation naphtha[3] and a long-handle brush, then repeat with acetone in place of the naphtha. Fill the test tubes with aqua regia and allow to stand for 24 hr. or more at room temperature. Rinse with tap water until acid is removed and follow with acetone to remove organic reaction products. If, at this point, a ring deposit is left around the inside of the test tubes at the normal air-oil interface, rinse with hydrofluoric acid (approximately 50 per cent). Allow the hydrofluoric acid to remain in the test tubes only long enough to disintegrate the deposit without necessarily dissolving it, then immediately flush with tap water. Complete the cleaning by following the procedure described in Paragraph (*a*).

(*c*) *Used Oxygen Delivery Tubes.*—Do not re-use oxygen delivery tubes because of the difficulty of cleaning fritted glass.

(*d*) *Used Condensers.*—Clean used condensers as described in Paragraph (*a*).

Procedure

6. (*a*) Slide the catalyst coils over the oxygen delivery tube resting one end on the fritted-glass outlet and place in the test tube. Pour 300 ml. of the oil sample over the coils into the test tube, wetting the coils completely with the oil, and then immerse the test tube in the warm (Note 2) oil bath to a depth such that the oil in the bath is at least 75 mm. (3 in.) above the level of the oil sample in the test tube. Then place the condenser over the oxygen delivery tube and test tube and make connections to the cooling water. (The temperature of the outlet water should not exceed 35 C. (95 F.) at any time during the test.) Connect the oxygen delivery tube to the oxygen supply through the flow meter.

NOTE 2.—The oil bath should be adjusted beforehand to a temperature high enough to maintain the oil in the desired number of oxidation cells at the required temperature of 95 ± 0.5 C. (203 ± 0.9 F.). A bath temperature of approximately 97 to 98 C. (206 to 208 F.) is normal.

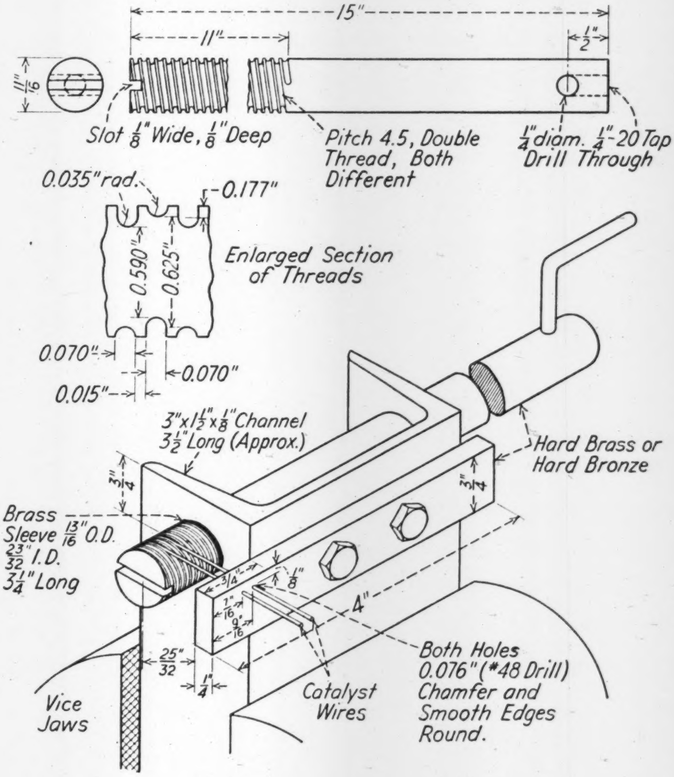
15"
11"
1/2"
11/16"
Slot 1/8" Wide, 1/8" Deep
Pitch 4.5, Double Thread, Both Different
1/4" diam. 1/4"-20 Tap Drill Through
0.035" rad.
-0.177"
0.590"
0.625"
Enlarged Section of Threads
0.070"
0.070"
0.015"
3"x1 1/2"x1/8" Channel 3 1/2" Long (Approx.)
Hard Brass or Hard Bronze
3/4"
3/4"
Brass Sleeve 13/16" O.D. 23/32" I.D. 3 1/4" Long
3/4"
1/8"
7/16"
9/16"
4"
25/32"
1/4"
Vice Jaws
Catalyst Wires
Both Holes 0.076" (#48 Drill) Chamfer and Smooth Edges Round.

FIG. 2.—Mandrel for Winding Catalyst Coils.

(*b*) Adjust the rate of flow of oxygen to 3 ± 0.5 l. per hr. and continue for 30 min., then add 60 ml. of distilled water at a temperature of approximately 95 C. (203 F.). Record the time when the water was added as the starting time of the test. During the test, adjust the rate of flow of oxygen, if necessary, at least twice a day to 3 ± 0.5 l. per hr.

(*c*) Check the temperature of the oil-water mixture once every hour for 3 hr. after the start of the test, or as often as necessary until a constant temperature of 95 ± 0.5 C. (203 ± 0.9 F.) is read twice in succession. After the constant temperature has been obtained, maintain the bath temperature constant and check it daily during the test.

(*d*) Add additional distilled water at a temperature of approximately 95 C. (203 F.) to the oxidation cell as required to maintain the water level at least 25 mm. (1 in.) above the fritted-glass outlet of the oxygen delivery tube.

(*e*) As soon as acid starts to form (Note 3) in the oil sample, withdraw oil from the cell at suitable intervals and determine the neutralization number in accordance with the Tentative Method of Test for Neutralization Number of Petroleum Products by Color-Indicator Titration (A.S.T.M. Designation: D 663)[5] or the Tentative Method of Test for Neutralization Number of Petroleum Products by Electrometric Titration (A.S.T.M. Designation: D 664)[5] of the American Society for Testing Materials. Continue these measurements until a neutralization number of at least 2.0 is reached.

(*f*) Plot the neutralization numbers as ordinates and time of duration of test as abscissas and read from the resulting curve the time required for the oil to reach a neutralization number of 2.0.

NOTE 3.—The presence of acid may be detected by a pronounced decrease of the interfacial tension between the oil sample and the distilled water or by the development of a distinctive odor of oxidized oil. A method for determining interfacial tension is described in the Appendix. Oil taken from a cell for an interfacial tension determination shall be returned to that cell within 2 hr.

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[5] 1942 Book of A.S.T.M. Standards, Part III, pp. 954, 958.

APPENDIX

DETERMINATION OF INTERFACIAL TENSION OF OIL AND WATER

Apparatus:

A1. The Cenco-du-Nouy interfacial tensiometer No. 70540 or equivalent apparatus is suitable.

Calibration:

A2. Calibrate the instrument and adjust the zero point according to the procedure furnished by the manufacturer.

Checking Purity of Water:

A3. (*a*) Before making the interfacial tension measurement, check the adjustment of the instrument and the purity of the water by measuring the surface tension of the water as follows:

(*b*) Clean a 100 to 150-ml. beaker by washing with hot chromic acid cleaning solution and rinsing thoroughly with distilled water. Add distilled water at a temperature of 25 ± 1 C. and place the beaker on the adjustable platform of the instrument. Clean the platinum ring by rinsing in A.S.T.M. precipitation naphtha[3] and heating in the oxidizing portion of a gas flame. Put the platinum ring in place and raise the adjustable platform until the ring is immersed in the water to a depth of not over 6 mm. Slowly lower the platform and maintain the torsion arm in the zero position by increasing the torque of the ring system. As the film of water adhering to the ring approaches the breaking point, proceed slowly with the adjustment to make certain that the moving system is in the zero position when rupture occurs.

(*c*) The scale reading at which the ring detaches from the water represents the uncorrected surface tension of the water. An uncorrected scale reading of 75 to 76 for a 6-cm. ring corresponding to a surface tension of 71 to 72 dynes per cm. indicates that the water is sufficiently pure for determining the interfacial tension of the oil and water. Low readings are caused by impurities in the water, improperly cleaned apparatus, or improper adjustment of the tensiometer.

Procedure:

A4. (*a*) Immerse the ring in the water to a depth of approximately 5 mm. Pour the oil to be tested, which has previously been filtered through an unwashed, medium filter paper[6] (150 mm. in diameter) using 1 filter per 25 ml. of oil used, over the water surface to a depth of approximately 10 mm. (Note 4).

NOTE 4.—If a ring with a short stirrup is used, it is recommended that the oil level be kept below the top of the stirrup to prevent bridging. If this is impossible, break the bridge oil film with a suitable clean, sharp object as soon as possible after the top of the stirrup is withdrawn from the oil. Care should be taken that the ring does not come in contact with the oil-water interface before beginning the interfacial measurement.

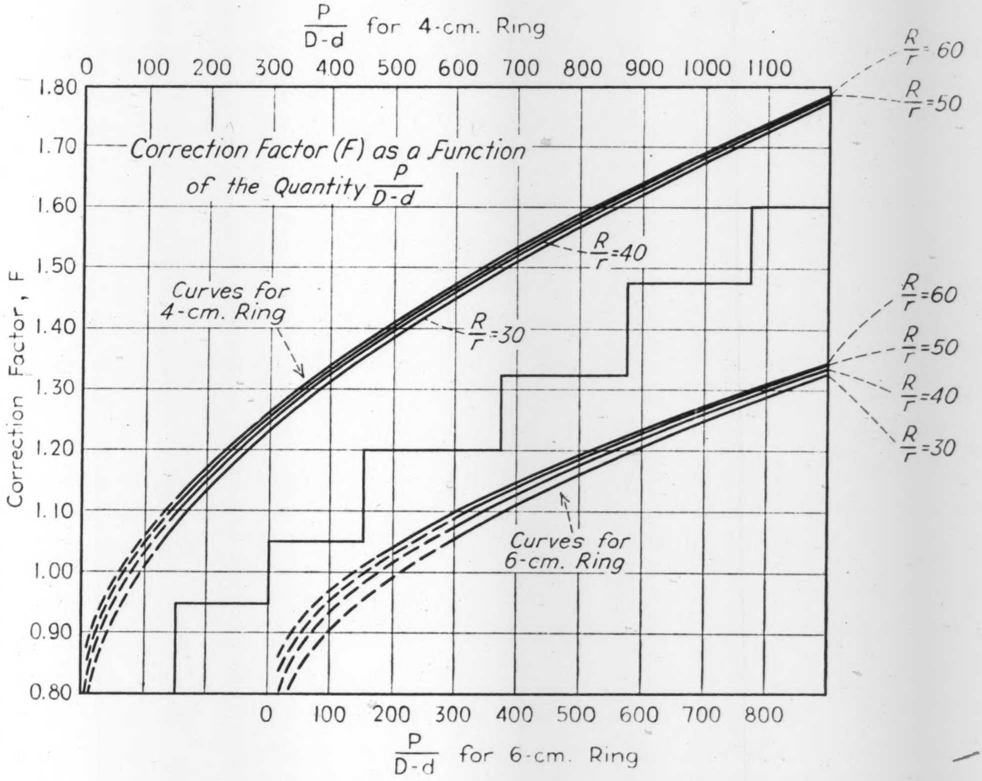

FIG. 3.—Correction Factor Curve for Interfacial Tension of Oil and Water.

(*b*) Allow the oil-water interface to age for 30 sec. Lower the platform and increase the torque of the ring system as before, so that, as nearly as possible, 30 sec. are required to draw the ring through the interface. As the breaking point is approached, proceed slowly, as the break is usually sluggish and too rapid movement of the torsion wire will result in a high reading.

(*c*) The scale reading is the uncorrected interfacial tension and should be corrected by applying the correction factor obtained from the curve in Fig. 3.

Calculation:

A5. The curve used for obtaining the proper correction factor is determined by the mean circumference of the ring and the ratio R/r, R and r being the radii of the ring and wire, respectively. (These values are furnished by the manufacturer of the ring.)

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[6] Whatman No. 1 filter paper or equivalent is satisfactory.

After selecting the proper curve to use, the value of the following expression shall be obtained from the experimental data:

$$\frac{P}{D-d}$$

where:

P = scale reading,

D = density of the water, and

d = density of the oil

Using the value of this expression, the corresponding value of F, the correction factor, shall be read from the correction curve. The corrected interfacial tension S shall be obtained by multiplying the scale reading P by the correction factor F.

REPORT OF SECTIONAL COMMITTEE

ON

PETROLEUM PRODUCTS AND LUBRICANTS

ASA PROJECT: Z 11

Sectional Committee Z11 on Petroleum Products and Lubricants held one meeting during the year: in Atlantic City, N. J., on June 23, 1942.

During the past year, the American Standards Association, on the recommendation of the sectional committee, reapproved four American Standards in which revisions have been made, and likewise approved two A.S.T.M. standards as American Standard for the first time, assigning the following ASA designations:

A.S.T.M. Standard Methods of:

Test for Melting Point of Paraffin Wax (D 87 - 42; ASA Z11.4-1942),
Test for Flash Point by means of the Pensky-Martens Closed Tester (D 93 - 42; Z11.7-1942),
Test for Gum Content of Gasoline (D 381 - 42; Z11.36-1942),
Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 42; Z11.44-1942),
Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 - 42; Z11.47-1942), and
Test for Tetraethyl Lead in Gasoline (D 526 - 42; Z11.48-1942).

At its meeting held during this Annual Meeting, the following two tentative standards were recommended by Committee D-2 on Petroleum Products and Lubricants for reference to letter ballot of the Society for adoption as standard. The sectional committee voted to recommend to the American Standards Association the approval of these methods as American Standard upon their adoption as A.S.T.M. standards:

A.S.T.M. Tentative Methods of:

Test for Carbonizable Substances in White Mineral Oil (Liquid Petrolatum) (D 565 - 41 T), and
Test for Carbonizable Substances in Paraffin Wax (D 612 - 41 T).

On the recommendation of Committee D-2, the sectional committee also voted to submit the Standard Method of Test for Dropping Point of Lubricating Grease (D 566 - 42) to the American Standards Association for approval as American Standard.

These three recommendations will accordingly be referred to letter ballot in the sectional committee.

Since the standards listed below, already having ASA approval, have been revised this year, Committee D-2 has recommended that they be presented to the ASA in their revised form for reapproval as American Standard:

Revision of A.S.T.M. Standard Methods of:

Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 42; Z11.44-1942), and
Viscosity-Temperature Charts for Liquid Petroleum Products (D 341 - 39; Z11.39-1939).

The sectional committee voted to refer these revisions to the ASA, subject to approval by letter ballot.

Respectfully submitted on behalf of the sectional committee,

T. A. Boyd,
Chairman.

R. E. Hess,
Secretary.

REPORT OF COMMITTEE D-3

ON

GASEOUS FUELS

A joint meeting of Committee D-3 on Gaseous Fuels and its seven subcommittees was held in Chicago, Ill., on October 4, 1942.

At this meeting the selections of the nominating committee for officers and members of the Advisory Committee were unanimously elected. Reports were presented by subcommittee chairmen on the work of their groups for the past year. Much progress has been made by the subcommittees in spite of curtailment of many activities in view of pressure of war work. The various projects under way are of an essential nature due to their importance in various phases of the war effort. Assistance has been derived in certain specific cases from studies already completed under committee sponsorship.

Brief accounts of the activities of various subgroups for the year are given below.

Subcommittee III on Determination of Calorific Value of Gaseous Fuels (R. B. Harper, chairman).—During the current year all controversial matters have been cleared up relative to a method for the determination of the heating value of gases by use of a flow type calorimeter. Material is being placed in a suitable form for distribution to the members of the subcommittee. It is expected that further consideration and perhaps approval of the method will be given in the near future.

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (E. F. Schmidt, chairman).—A very complete report covering work by the National Bureau of Standards on 11 specific gravity instruments submitted by their manufacturers was issued during 1942. It was supplied to all members of the supervising subcommittee as well as to members of Committee D-3. While it was planned to publish a summary of this very complete study as soon as the necessary material could be prepared, this has not as yet been possible due to pressure of war work. In view of the importance of the information contained, it was decided during the year to make copies available to members of all subcommittees desiring it. As a result a number of additional copies were so distributed.

It is hoped that arrangements may shortly be completed for publishing a summary of this very valuable report within the next few months.

Subcommittee V on Determination of Special Constituents of Gaseous Fuels (E. F. Pohlmann, chairman).—An investigation is now in progress at the Institute of Gas Technology, Chicago, Ill., on the identity and proportions of specific sulfur compounds in gaseous fuels. One investigator is devoting his full time to the preparation of analytical methods covering determination of total organic sulfur, carbon disulfide, carbon oxysulfide, mercaptans, and thiophenes.

Methods for the determination of these various constituents will probably be ready late in 1943.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels. (A. W. Gauger, chairman).—Some objections were raised by members of the committee to an improved portable colorimeter previously developed which employed change in color of cobaltous bromide with water. These were based on its cost as well as the necessity of its manufacture especially for this type of work. Studies therefore were made on measurement of color change by means of some suitable commercial colorimeter. The Evelyn colorimeter has proved ideal for the purpose because of its simplicity of operation and adaptability for portable use. It is necessary to take readings at a narrow spectral band, 660 $m\mu$, and to hold the solutions to a selected constant temperature, reproducible to ± 0.1 F. Success of laboratory experiments with this equipment seemed to warrant certain field tests. Natural gas field samples were taken and their percentage of water vapor determined by means of the colorimeter and by the dew-point method. In general, results were highly successful.

Efforts to find less expensive methods for determining water content of gases led also to a preliminary examination of the so-called Crismer test or cloud method. Two partially miscible liquids, isopropyl alcohol and a mixture of 50 per cent of white oil and 50 per cent of kerosine, are mixed in proportion of 6.3 and 3.7 ml., respectively, and the cloud point observed. This is raised about 12 C. per 1 per cent water added to the alcohol. Sensitivity of the test as indicated on synthetic mixtures of water in alcohol shows a reproducibility of plus or minus 1 per cent error or less in water concentrations between 1 and 2 per cent. Twenty samples of air saturated at 20 C. were frozen and analyzed by the Crismer test. These showed an average error of plus or minus 2 per cent from the amount of water determined in a drying tube.

Another attempt to develop a simple method for measurement of water content of gas utilizes calcium carbide, 2 moles of water reacting to produce 1 mole of acetylene. The change was studied both by constant pressure and constant volume methods. Changes in both pressure and volume, however, are so small that it is questionable whether a simple apparatus yielding the precision required could be devised. These methods, therefore, are considered impracticable.

This report has been submitted to letter ballot of the committee which consists of 33 members; 28 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. W. GAUGER,
Chairman.

R. M. CONNER,
Secretary.

REPORT OF COMMITTEE D-4
ON
ROAD AND PAVING MATERIALS

Committee D-4 on Road and Paving Materials held one meeting during the year, in Atlantic City, N. J., on June 23, 1942.

Subsequent to the 1942 Annual Meeting, Committee D-4 presented to the Society through Committee E-10 on Standards, the following recommendations:

New Tentative Specifications for:

Crushed Stone, Slag, and Gravel for Bituminous Concrete Base and Surface Courses of Pavements (D 692 - 42 T), to replace immediately Specifications D 485 - 38 T, D 486 - 39 T, and D 309 - 30,

Crushed Stone and Crushed Slag for Bituminous Macadam Base and Surface Courses of Pavements (D 693 - 42 T), to replace immediately Specifications D 192 - 41 T and D 487 - 42 T.

Crushed Stone, Slag, and Gravel for Water-Bound Base and Surface Courses of Pavements (D 694 - 42 T), to replace immediately Specifications D 488 - 38 T and D 489 - 40 T,

Revision of Tentative Specifications for:

Standard Sizes of Coarse Aggregate for Highway Construction (D 448 - 41 T), and

Revision of Tentative Methods of:

Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75 - 40 T).

These recommendations were accepted[1] by Committee E-10 on August 24, 1942, and the new and revised tentative standards appear in the 1942 Book of A.S.T.M. Standards, Part II.

The proposed Method of Test for Moisture-Density Relations of Soils, submitted jointly by Committee D-4 and Committee D-18 on Soils for Engineering Purposes were accepted[1] by Committee E-10 on September 17, 1942, for publication as tentative. This new tentative method was assigned the designation D 698 - 42 T and appears in the 1942 Book of A.S.T.M. Standards, Part II.

I. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative specifications be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Slow-Setting Emulsified Asphalts (for Fine Aggregate Mixes) (D 631 - 41 T),

Asphalt Filler for Brick Pavements (D 241 - 39 T), and

Sodium Chloride (D 632 - 41 T).

II. STANDARDS AND TENTATIVE STANDARDS CONTINUED WITHOUT REVISION

Individual subcommittees of Committee D-4 have reviewed the various specifications and methods of test that have been published without revision for a number of years and have no changes to recommend other than those previously covered in this report. Committee D-4 therefore approves retaining these standards and tentative standards in their present form without revision.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-4 reported the following results of the letter ballot vote from a committee membership of 129: *D 692 - 42 T.*—This is a revision and consolidation of three tentative specifications upon which the vote was as follows: D 485, affirmative 67, negative 0, ballots marked "not voting" 15; D 486, affirmative 69, negative 0, ballots marked "not voting" 13; D 309, affirmative 69, negative 1, ballots marked "not voting" 12. *D 693 - 42 T.*—This is a consolidation of two existing specifications made as an editorial change to conform with the combination effected by D 692. *D 694 - 42 T.*—This is a revision and consolidation of two tentative specifications and new specifications for gravel upon which the vote was as follows: D 488 and D 489, affirmative 67, negative 0, ballots marked "not voting" 15; new specifications for gravel, affirmative 68, negative 1, ballots marked "not voting" 13. D 448 - 42 T, affirmative 71, negative 0, ballots marked "not voting" 11. D 75 - 42 T, affirmative 69, negative 2, ballots marked "not voting" 11. D 698 - 42 T, affirmative 67, negative 1, ballots marked "not voting" 14.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Slow-Setting Emulsified Asphalts (for Fine Aggregate Mixes) (D 631 – 41 T)	69	2	17
Spec. for Asphalt Filler for Brick Pavements (D 241 – 39 T)........................	62	1	11
Spec. for Sodium Chloride (D 632 – 41 T).............	70	0	15

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 128 members with the results shown in Table I.

This report has been submitted to letter ballot of the committee which consists of 128 members; 88 members returned their ballots, of whom 81 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

C. N. FORREST,
Chairman.

PRÉVOST HUBBARD,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-4 presented to the Society through Committee E-10 on Standards the following recommendations:

Tentative Revision of Standard:

Specifications for Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 544 – 41), and

Methods of Testing Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 545 – 41).

These recommendations were accepted by Committee E-10 on November 4, 1943, and the tentative revisions appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part II, pp. 164 and 165.

On November 10, 1943, Committee E-10 accepted proposed revisions in the Tentative Specifications for Tar (D 490 – 38 T) recommended by Committee D-4. The revised tentative specifications appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part II, p. 112, bearing the designation D 490 – 43 T.

REPORT OF SECTIONAL COMMITTEE
ON
ROAD AND PAVING MATERIALS
ASA PROJECT: A37

This is the first formal report on behalf of Sectional Committee A37 on Road and Paving Materials since its reorganization in 1941. Prior to this reorganization, the personnel of the sectional committee was coincident with that of the Society's Committee D-4 on Road and Paving Materials. This caused the personnel to be rather large and unwieldy and to lose much of the representative nature which a sectional committee functioning under the procedure of the American Standards Association should have. It was accordingly reorganized with a more restricted personnel consisting of representatives of the following organizations:

American Association State Highway Officials,
American Concrete Institute,
American Petroleum Institute,
American Public Works Association,
American Road Builders Association,
American Society of Civil Engineers,
American Society for Testing Materials,
American Transit Association,
Asphalt Institute,
Associated General Contractors of America,
Association of Asphalt Paving Technologists,
Calcium Chloride Association,
Concrete Reinforcing Steel Institute,
Federal Works Agency, Public Roads Administration,
Highway Research Board,
National Bureau of Standards,
National Crushed Stone Association,
National Paint, Varnish, and Lacquer Association,
National Paving Brick Association,
National Sand and Gravel Association,
National Slag Association,
Portland Cement Association,
U. S. Army, Office of the Chief of Engineers,
U. S. Dept. of Commerce, Civil Aeronautics, and
U. S. Navy, Bureau of Yards and Docks.

The revised personnel was approved by the ASA on May 21, 1942.

In reorganization, the scope of the committee was also revised somewhat so as to include specifications as well as methods of testing whereas the previous scope was limited to methods of testing alone. The revised scope reads as follows:

> SCOPE.—Specifications, methods of test, and nomenclature relating to road and paving materials, excepting hydraulic cements.

Meetings of the committee were held in Chicago, Ill., on June 25, 1941 (reorganization meeting), and in Cleveland, Ohio, on March 4, 1942. The A37 Executive Committee met in Atlantic City, N. J., on June 24, 1942.

The following A.S.T.M. standards, submitted to the American Standards Association on the recommendation of Sectional Committee A37, were approved as American Standard during the year:

A.S.T.M. Standard Methods of Test for:

Determination of Bitumen (A.S.T.M. D 4 - 42; ASA A37.3—1943),
Amount of Material Finer than No. 200 Sieve in Aggregates (C 117 - 37; A37.4—1943),
Specific Gravity and Absorption of Coarse Aggregate (C 127 - 42; A37.5—1943),
Specific Gravity and Absorption of Fine Aggregate (C 128 - 42; A37.6—1943),

Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 - 39; A37.7—1943),
Sieve Analysis of Fine and Coarse Aggregates (C 136 - 39; A37.8—1943),
Distillation of Tar Products Suitable for Road Treatment (D 20 - 30; A37.9—1943),
Softening Point of Bituminous Materials (Ring-and-Ball Method) (D 36 - 26; A37.10—1943),
Ductility of Bituminous Materials (D 113 - 39; A37.11—1943),
Proportion of Bitumen Soluble in Carbon Tetrachloride (D 165 - 42; A37.12—1943),
Residue of Specified Penetrations (D 243 - 36; A37.13—1943), and
Sieve Analysis of Mineral Filler (D 546 - 41; A37.14—1943).

The committee plans to review and compare the existing standards in the field of road and paving materials. If gaps are found or it is felt there is a need for correlation, the matter will be referred back to the originating group which will also be advised of any differences. It may be that steps will need to be taken to see what substitutes can be found in the case of any specifications that cover a material that is particularly difficult to obtain.

Respectfully submitted on behalf of the sectional committee,

H. F. CLEMMER,
Chairman.

R. E. HESS,
Secretary.

REPORT OF COMMITTEE D-5

ON

COAL AND COKE

Committee D-5 on Coal and Coke held a meeting in Buffalo, N. Y., on March 1, 1943; another meeting will be held at the annual meeting of the Society in June, 1943, in Pittsburgh, Pa. Subcommittee XIII on Coal Sampling, Subcommittee XV on Plasticity and Swelling of Coal, and Subcommittee XVI on Ignitibility of Coal and Coke held meetings in St. Louis, Mo., on September 29, 1942; and on March 1, 1943; meetings of Subcommittee I on Methods of Testing, and of Subcommittees XIII and XV were held in Buffalo, N. Y.

A new Method of Test for Free-Swelling Index of Coal[1] is recommended for publication as tentative. Six methods under investigation for determination of plastic properties of coal, including expansion and expansion pressures developed during carbonization, are published as information as Appendices to this report. The Tentative Method of Sampling Coals Classed According to Ash Content (D 492 - 40 T) has been revised extensively[2] to include sampling of large lump coal and run-of-mine coal, and a procedure for sampling coal for determination of total moisture has been included. The tentative revisions of Sections 27 to 31 of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 42), pertaining to the determination of the fusibility of ash, are recommended for adoption as standard with some further revisions. A statement has been prepared for insertion in these standard methods calling attention to changes in composition of coal samples on standing caused by oxidation, making it essential that such samples be analyzed as soon as possible after collection and placing a limit of 30 days between sampling and analysis of coal when the Btu. determination is of importance. A tentative revision of Standard Method D271 is also submitted.

There appears under the activities of subcommittees brief explanations concerning these recommendations.

A paper by G. C. Soth and C. C. Russell entitled "The Gieseler Method for Measurement of the Plastic Characteristics of Coking Coal"[3] is being presented at the annual meeting of the Society. The Gieseler method is one of six methods for evaluating plastic properties of coals now under investigation by Subcommittee XV.

During the year H. C. Dickinson of the National Bureau of Standards resigned his membership on the committee because of the necessity of curtailing some of his activities. Since it is desirable to have a representative of the National Bureau of Standards on the committee, in view of the importance of the calorimetric determination in coal analysis, R. S. Jessup has been appointed as the representative of the Bureau. Mr. Lowell Malan, Chief Chemist, The United Electric Coal Co., Chicago, Ill., has been appointed a member of the committee.

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[1] This method was accepted as tentative by the Society and appears in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 154.

[2] This revised method was accepted as tentative by the Society and appears in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 147.

[3] See p. 1176.

The present membership of Committee D-5 is 54, of whom 15 are classified as producers, 16 as consumers, and 23 as general interest members.

I. NEW TENTATIVE STANDARD

The committee recommends that the Method of Test for Free-Swelling Index of Coal,[1] prepared by Subcommittee XV, be accepted for publication as tentative.

II. REVISION OF TENTATIVE STANDARD

The committee recommends that the Tentative Method of Sampling Coals Classed According to Ash Content (D 492 - 40 T) be revised as appended hereto,[2] and continued as tentative, as proposed by Subcommittee XIII.

III. REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions prepared by Subcommittee I for inclusion in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 42)[4] as set forth below and accordingly asks for a nine-tenths affirmative vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Section 4 (a).—Change to read as follows by addition of the italicized words:

4. (*a*) Spread the sample on tared pans, weigh, and air-dry at room temperature, or in the special drying oven shown in Fig. 1 at 10 to 15 C. above room temperature, and weigh again. Continue the drying until the loss in weight is not more than 0.1 per cent per hr. *Drying should not be continued beyond this point because of the oxidation of the coal.* Complete the sampling as described in Section 3 for dry coal.

Section 5 (b).—Change to read as follows by addition of the italicized words:

5. (*b*) Spread the sample on tared pans, weigh, and air-dry in the special moisture oven shown in Fig. 1 at 10 to 15 C. above room temperature until the loss in weight between two successive weighings made 6 to 12 hr. apart does not exceed 0.1 per cent per hr. *Drying should not be continued beyond this point because of the oxidation of the coal.* Record the loss in weight as "air-drying loss."

New Section.—Add a new Section 44 at the end of the methods to read as follows:

44. Deterioration of Coal Samples.—Since coal samples on standing oxidize and change in composition, it is essential that they be analyzed as soon as possible after collecting the gross samples. If the Btu. determination is an important consideration in the sale of coal, the time elapsed between sampling and analysis shall not exceed 30 days.

IV. TENTATIVE REVISION OF STANDARD

The committee recommends for publication as tentative the following revision in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 42):

Section 11.—Add the following note to this section:

NOTE 3.—Difficulty may be experienced in securing satisfactory check determinations of ash in the same or different laboratories for coals unusually high in calcite and pyrite. This is caused by varying amounts of sulfate sulfur being retained in the ash. When such difficulty is encountered, or when coals of relatively high ash content whose mineral matter composition is unknown are encountered, the ash should be determined by the following modified procedures:

(*a*) Place the porcelain capsules containing the dried coal from the moisture determination in a cold muffle furnace and heat gradually so that the temperature reaches 500 C. in 1 hr., and 750 C. in 2 hr. Heat to constant weight at 750 C. By this means pyritic sulfur will be oxidized and expelled before the calcite is decomposed. An ample supply of air in the muffle must be assured at all times to insure complete oxidation of the pyritic sulfur and proper circulation through the muffle must be assured to remove the SO_3 formed.

(*b*) The modified procedure described in Paragraph (*a*) should be adequate for determining ash in all troublesome commercial samples. However, samples may be encountered in certain special studies whose ash values are quite high and whose mineral matter contains much greater than normal amounts of calcite and pyrite. In such cases sulfate sulfur should be determined on the ash obtained by the modified cold muffle method and the value properly corrected, or the Parr[a] sulfated ash method as modified by Rees[b] should be used.

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[4] 1942 Book of A.S.T.M. Standards, Part III, p. 15.

V. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD

The committee recommends that the tentative revision of Sections 27 to 31[5] of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 42) be revised as follows, as proposed by Subcommittee I, and approved for reference to letter ballot of the Society for adoption as standard:

Section 27 (a).—Delete from Paragraph (*a*) the note which reads as follows:

> NOTE.—Commercial gas-fired furnaces which meet these requirements are the coal-ash fusion furnace of the Denver Fire Clay Co., the No. 3 Melter's Furnace of the American Gas Furnace Co. modified to provide an observation hole in the side, and the Barrett coal-ash fusion furnace of the Burrell Technical Supply Co.

Section 30 (b).—Change from its proposed revised form: namely,

> (*b*) A reducing atmosphere shall be maintained surrounding the cones during the heating. The ratio by volume of reducing gases to oxidizing gases in this atmosphere shall be between the limits 20 to 80, and 70 to 30 and preferably near the value 40 to 60.[6] Hydrogen, hydrocarbons, and carbon monoxide shall be considered as reducing gases, and oxygen, carbon dioxide, and water vapor shall be considered as oxidizing gases. At least 40 per cent of the reducing gases shall be hydrogen. With gas-fired furnaces so constructed that the combustion gases have free access to the ash cones, the required reducing atmosphere can be maintained by operating the furnace with an excess of gas.

to read as follows:

> (*b*) A mildly reducing atmosphere shall be maintained surrounding the cones during the heating. The ratio by volume of reducing gases to oxidizing gases in this atmosphere shall be between the limits 20 to 80 and 80 to 20.[6] Hydrogen, hydrocarbons, and carbon monoxide shall be considered as reducing gases and oxygen, carbon dioxide, and water vapor shall be considered as oxidizing gases. Nitrogen is inert.

VI. PUBLICATION AS INFORMATION

The following six proposed methods prepared by Subcommittee XV are presented for publication as information as Appendices to this report:

Proposed Methods of Test for:

- Expansion Pressure of Coal During Coking,
- Plastic Properties of Coal by the Davis Type Plastometer,
- Plastic Properties of Coal by the Giesler Type Plastometer,
- Carbonization Pressure of Bituminous Coal,
- Measurement of Pressures Developed During Carbonization of Coal by the Movable Wall Oven,
- Expansion Properties of Coal for Use in By-Product Coke Ovens by Means of the Bethlehem Test Oven,

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 54 members; 48 members returned their ballots, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Testing (W. A. Selvig, chairman) recommended statements for inclusion in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 42) regarding the oxidation of coal samples by excessive air-drying and setting a limit of 30 days on the permissible time elapsed between sampling and analysis of coal if the Btu. is an important consideration. It is not good laboratory practice to prolong unnecessarily the time of air-drying coal samples, as this will cause some oxidation with change in composition. Likewise, the storage of coal samples for unnecessarily long periods before analysis should be avoided as there will be a loss in Btu. on account of oxidation.

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[a] S. W. Parr, "Chemical Study of Illinois Coal," Bulletin No. 3, p. 35, Illinois Coal Mining Investigations, State Geological Survey, Urbana, Ill. (1916).

[b] O. W. Rees, "Determining Ash in High Carbonate Coals. Study of the modified Method," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 9, pp. 307–309 (1937).

[5] 1942 Book of A.S.T.M. Standards, Part III, p. 1587.

[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 870 (1942).

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Test for Free-Swelling Index of Coal....................	41	1	6
II. REVISION OF TENTATIVE STANDARD			
Method of Sampling Coals Classed According to Ash Content (D 492 - 40 T)... ..	44	0	4
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 42; ASA K18-1942)..................	43	0	5
IV. TENTATIVE REVISION OF STANDARD			
Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 42; ASA K18-1942)..................	41	0	7
V. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 42; ASA K18-1942), as revised......	43	0	5

A Special Section on Uniformity of Analytical Data, under the chairmanship of H. H. Lowry, was discharged as it had completed its assignment with the presentation of the paper on "Statistical Study of the Precision of Methods for Analysis of Coal and Coke,"[6] by H. H. Lowry and Charles O. Junge, Jr., at the 1942 annual meeting of the Society. This study indicates that certain revisions may be desirable in some of the present tolerances between duplicate analyses of coal and coke, but the subcommittee is not ready at this time to recommend specific revisions of these tolerances.

Subcommittee XIII on Coal Sampling (H. F. Hebley, chairman) has been very active this past year and recommended extensive revisions in the Tentative Method of Sampling Coals Classed According to Ash Content (D 492 - 40 T).[2] This revision includes procedures for sampling large lump coal and run-of-mine coal, which sizes are difficult to sample properly. The subcommittee collected much factual information bearing on the sampling of large sizes of coal. Sampling procedures for total moisture are included in the method, and the formulation of these procedures required many sampling experiments, some of which were published.[7]

A special section of the subcommittee, consisting of W. D. Langtry, chairman, J. D. Doherty, H. F. Hebley, O. O. Malleis, and O. W. Rees has been appointed to consider special methods of sampling coal for the determination of volatile matter in connection with city smoke ordinances.

Subcommittee XV on Plasticity and Swelling of Coal (C. C. Russell, chairman) has recommended the Method of Test for Free-Swelling Index of Coal for publication as tentative. The method has been adapted to American practice from the British Standards Institution Method for Crucible Swelling Test for Coal. It is a small-scale laboratory test giving information concerning the free-swelling and coking properties of coal when burned as a fuel. It is not recommended as a test for determining expansion properties of coals when carbonized in coke ovens.

The subcommittee has under consideration six other methods for determining plastic properties of coals and and for evaluating their expansion and pressure characteristics in coke ovens during carbonization. To facilitate their use these test methods have been written up in A.S.T.M. form and are appended hereto as information. It is hoped that making them readily available will result in their greater use so they can be better evaluated. It is not the intention of the subcommittee that all of these methods necessarily be recommended eventually for publication as tentative standards. Probably some of the methods can be combined into single procedures. Eventually the most promising of the methods can be selected for publication as tentative.

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[7] H. M. Cooper, N. H. Snyder, and R. F. Abernethy, "Moisture Losses in Sampling Coal," *Report of Investigations 3670*, U. S. Bureau of Mines (1942).

This report has been submitted to letter ballot of the committee which consists of 54 members; 48 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. FIELDNER,
Chairman.

W. A. SELVIG,
Secretary.

APPENDIX I

PROPOSED METHOD OF TEST FOR EXPANSION PRESSURE OF COAL DURING COKING[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method[2] of test is intended for determining the relative expansion pressures exerted on oven walls by different coals during coking. The operating conditions for all coke ovens are not the same; the thickness of the charge and the temperature of the flues vary in different plants. Therefore, in this method, provision has been made for heating at low, medium, and high temperatures and also for varying the thickness of the charge from 5 to 13 in. The procedure described in the method covers only the 10-in. charge which shall be considered as standard practice.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Electric Furnace.*—An electric furnace, as shown in Fig. 1, with a movable wall mounted on a carriage with ball-bearing wheels running on standard mine car track, and suitably braced with angle and channel bars to withstand the coking pressure of the charge. The heating chamber shall be rectangular, 28 in. in width by 44 in. in height. The thickness may vary in accordance with the position of the movable wall. Both movable and fixed walls shall be heated by helices of No. 8 nickel-chromium wire placed horizontally in open slots of the refractory so that heating is for the most part by radiation. The helices shall be pulled apart in the middle of the chamber and drawn closer together at the sides so that heating is uniform over the walls. The end of the tube supporting the movable wall shall be fitted with a pressure gland, the piston of which shall be in contact with a strain screw passing through the saddle piece shown in Fig. 1. Steel rods provided with nuts shall pass through the saddle piece and fit into slots in the bracing irons on opposite sides of the furnace. The fixed and movable walls shall be enclosed in suitable steel casings and the space between the refractories and the casings shall be filled with diatomaceous earth and insulating brick. A scale, graduated in 0.01 in., shall be fixed to the side of the track and shall be read by a telescope. Replaceable sheets of No. 16 gage stainless steel shall be provided for covering the heating walls during the test.

(*b*) *Reversible Side Walls.*—Reversible side furnace walls 4 in. in thickness, made of insulating brick to minimize heat penetration into the sides of the charge. The preliminary heating of the chamber before charging shall be done with one side of the reversible walls exposed to radiant heat from the front and back walls; immediately before charging the side walls shall be reversed.

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[1] This proposed method is under the juristiction of the A.S.T.M. Committee D-5 on Coal and Coke. Published as information, June, 1943.

[2] For information concerning the experimental work on which this method is based, see H. S. Auvil, J. D. Davis and J. T. McCartney, "Expansion of Coal During Coking," U. S. Bureau of Mines, *Report of Investigations 3451*, 1939; and James T. McCartney, and Joseph D. Davis, "Expansion of Coal During Coking," U. S. Bureau of Mines, *Report of Investigations 3644*, 1942.

(*c*) *Sheet-Steel Retorts.*—Retorts made of No. 20 gage mild sheet steel, 27 in. in width by 44 in. in height and varying from 5 to 13 in. in thickness. The bottoms and tops of these retorts shall be made of No. 8 gage mild steel. Centrally through the top of each retort a 2-in. standard pipe offtake shall be welded. The retorts shall be fabricated by welding all seams except that the tops shall be left open for charging, being welded in place just prior to the test. Unless the rate of coking in the ovens where the coal is to be used is very rapid or very slow, the 10-in. retort shall be considered standard.[3]

(*d*) *Regulating Transformers.*—Regulating transformers with at least 20 voltage steps in the secondary so that the separate heating circuits can be supplied with approximately the current required (see Fig. 2).

(*e*) *Program Controller.*—A two-point thermoelectric recorder-controller designed to make and break the heating circuits separately and provided with mechanism to control the wall temperatures according to the time-temperature program selected (see Fig. 2).

(*f*) *Constant-Volume Controller.*—A constant-volume controller consisting of a reversible motor, reducing gears, suitable electric relays, chains and sprockets, and electrical contact points. The strain screw shall be driven to the right or left automatically as the pressure on the walls increases or decreases and the carriage shall move only through the distance between the contact points located between the carriage and track frames. This distance shall be adjustable to 0.006 in.

(*g*) *Recording Gage.*—A recording pressure gage to give a continuous record of the pressures on the furnace walls.

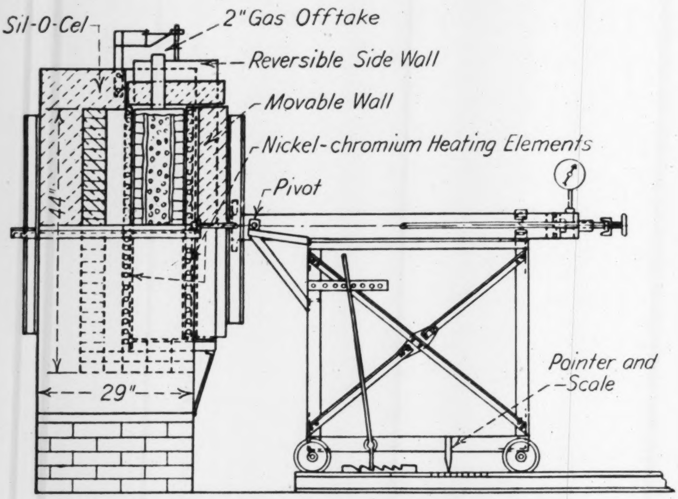

FIG. 1.—Electric Furnace with Movable Wall.

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[3] The 10-in. retort has been adopted tentatively by the Bureau of Mines as standard.

(*h*) *Retort Holder.*—Wooden bracing frames placed against the two sides of the retort and clamped together with tie rods, to prevent excessive bulging of the sides of the retort during charging.

Sample

3. The gross sample shall weigh approximately 4000 lb. and shall be crushed to 0 to $\frac{1}{4}$-in. size. The sample shall be obtained by riffling at least 670 lb. of coal from the gross sample. Samples for analysis and plastic tests shall be riffled from the 670-lb. sample, which shall then be cut in half to provide two samples of about 330 lb. each for duplicate determinations of expansion pressure.

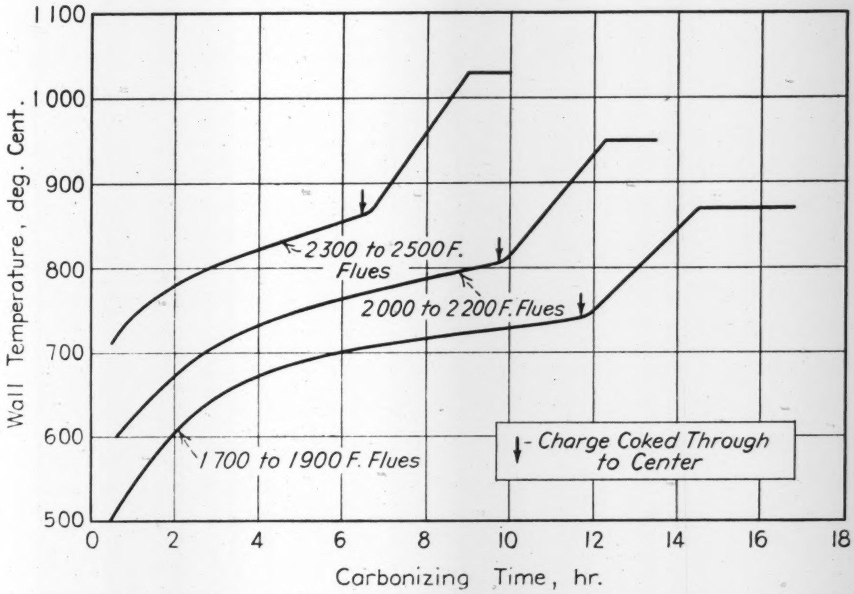

FIG. 2.—Wall Temperatures for Movable-Wall Electric Furnace Corresponding to Various Average Flue Temperatures.

Procedure Using Standard 10-in. Retort[3]

4. (*a*) *Charging the Retort.*—Two metal straps $\frac{1}{16}$ by $\frac{1}{4}$ in. shall be soldered inside the retort between the side walls to reduce bulging after the retort holder is removed. The retort shall be placed in the holder and the tie rods inserted and tightened. The weighed sample of coal (about 330 lb.) shall be placed in the retort leaving approximately 2 in. of top void space, and the top of the retort sealed in place by electric welding.

(*b*) *Preparing Furnace and Introducing Retort.*—The stainless steel sheets, which will have warped by cooling from a previous test temperature, shall be put in place and the furnace heated to approximately 1600 F. The sheets shall be flattened by advancing the movable wall against the fixed wall and applying a total pressure of about 2700 lb., after which the furnace shall be allowed to cool to approximately the point of starting the test, as shown by the curves in Fig. 2. The movable wall shall be moved back, the insulating side walls reversed, and the retort removed from the holder and lifted into place by a chain hoist and crane. A cover of insulating refractory material 5 in. in thickness shall be placed on top of the retort and the gas offtake connected to a pipe leading to the hood.

(*c*) *Adjustments.*—A load of 2700 lb. shall be applied to the retort to flatten the heating walls, which may have bulged slightly. After this adjusting period of not longer than 30 min., the applied load shall be reduced to 500 lb., and the constant-volume controller adjusted to maintain the coal charge at constant thickness for the remainder of the test. The program controller shall be set to follow the desired curve (see Fig. 2) and the test allowed to continue virtually automatically until a sudden drop in the gas rate indicates complete carbonization.

Reproducibility

5. Maximum pressures for duplicate determinations should agree within 0.3 psi. Maintenance of the same charge density in duplicate determinations is an important detail. For this method the average density should be about 50 lb. per cu. ft. in routine determinations, but it may be adjusted to other values if desired.

NOTE 1: *Gas Pressures at Center of Charge.*—If these pressures are much higher for a given determination than for the duplicate test, the wall pressures are likely not to agree. Measurement of gas pressures is fraught with uncertainty because of the likelihood of plastic coal getting into the pressure tubes and carbonizing there, giving rise to pressures that are not representative. Pressures should be measured at two or more points in the plane midway between the heated walls of the retort; if on release of the pressure at one point pressures at the other points drop, this is proof that there is no stoppage of the pressure tubes.

NOTE 2.—Use of stainless steel sheets between the heating walls and the retort is necessary because otherwise the iron oxide formed on the retort will form slag with the refractories and ultimately ruin them.

APPENDIX II

PROPOSED METHOD OF TEST FOR PLASTIC PROPERTIES OF COAL BY THE DAVIS TYPE PLASTOMETER[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test is intended for determining the relative plastic behavior of coal when heated under prescribed conditions in the absence of air.[2] The test is useful in predicting the coking behavior of bituminous coals and blends and for determining the extent of weathering of coal when results for the fresh coal are known.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Retort.*—A retort assembly consisting of six pieces as shown in Fig. 1. The retort proper shall be constructed of mild steel 5 in. in length, $1\frac{7}{8}$ in. in outside diameter, and a wall-thickness of $\frac{3}{16}$ in. It shall be provided with two screw-in cap head pieces. A $\frac{1}{4}$-in. hole shall be drilled through the center of the rear head piece and a thermocouple well 0.234 in. in outside diameter and $\frac{11}{64}$ in. in inside diameter shall be welded in this opening and shall extend inward to near the center of the retort. A tubular axle $\frac{7}{16}$ in. in outside diameter and $\frac{1}{4}$ in. in inside diameter shall be welded at the center of, and perpendicular to, the outside face of the rear head piece. This tube shall extend outward through a plain brass bearing and serves also as an extension of the thermocouple well. A hole $\frac{29}{64}$ in. in diameter shall be drilled through the center of the front head piece and a tubular axle $\frac{5}{8}$ in. in outside diameter and $\frac{29}{64}$ in. in inside diameter shall be welded perpendicularly around this hole. This axle shall extend outward from the retort and shall be fitted on the outer end with a sprocket wheel drive. A longer tubular shaft $\frac{7}{16}$ in. in outside diameter and $\frac{3}{16}$ in. in inside diameter shall pass through the $\frac{5}{8}$-in. diameter axle and one end shall be reamed out to fit around the thermocouple well with a clearance of 0.008 in. The other or front end of this shaft shall extend outward from the retort through a plain brass bearing and shall be fitted with a pulley carrying a dial. Four T-shaped rabble arms shall be fitted to the part of the shaft inside of the retort and spaced at 90-deg. intervals around the shaft. The horizontal bar of each T-shaped rabble arm shall be 1 in. in length and the threaded vertical bar of the T shall be of such length that when screwed into a collar on the shaft it leaves a clearance of 0.008 in. between the rear edge of the rabble arm and the inside wall of the retort. Each rabble arm shall be beveled slightly on its front or driving edge and on its top side or outside end to give a clearance slightly more than 0.008 in. between the beveled surface and the inside wall of the retort. A hexagonal nut shall be welded on the outside face of each of the two head pieces and around their axles. These nuts serve the double purpose of reinforcement and provision for using a wrench to open the retort after a test.

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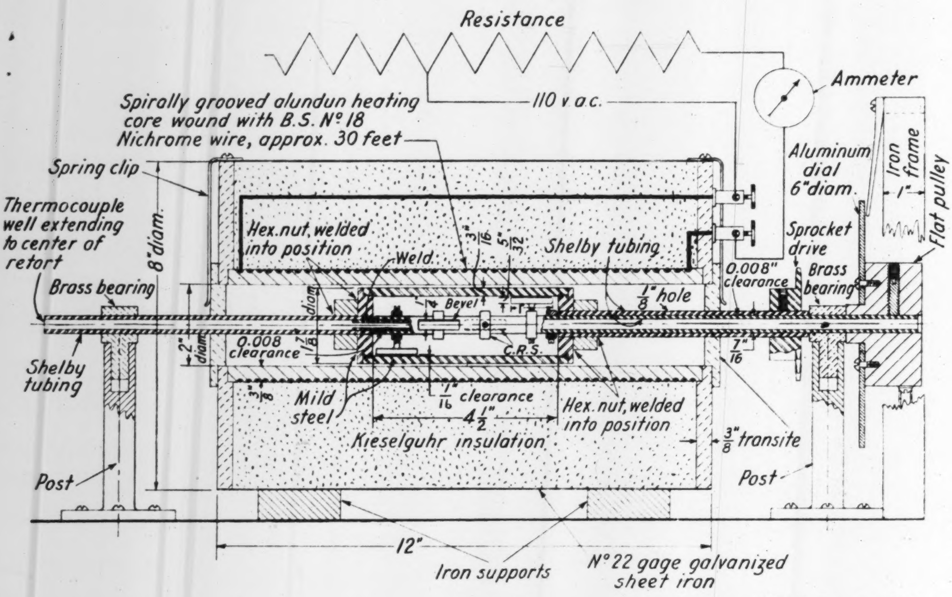
[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke. Published as information, June, 1943.

[2] For information concerning the experimental work on which this method is based, see the paper by R. E. Brewer and R. G. Atkinson, "Plasticity of Coals. Its Measurement and Relation to Quality of Coke Produced," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 8, p. 443 (1936); for further work, see R. E. Brewer and J. E. Triff, "Measurement of Plastic Properties of Bituminous Coals. Comparison of Gieseler and Davis Plastometer and Agde-Damm Dilatometer Methods," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 11, p. 242 (1939).


Resistance
110 v.a.c.
Ammeter
Spirally grooved alundun heating core wound with B.S. Nº 18 Nichrome wire, approx. 30 feet
Spring clip
Thermocouple well extending to center of retort
8" diam.
Brass bearing
Shelby tubing
2" diam.
Hex. nut, welded into position
Weld
$\frac{3}{16}$"
$\frac{5}{32}$"
Shelby tubing
Bevel
$1\frac{7}{8}$" diam.
0.008 clearance
C.R.S.
$\frac{1}{8}$" hole
0.008" clearance
$\frac{7}{16}$"
$\frac{3}{8}$"
Mild steel
$\frac{1}{16}$" clearance
$4\frac{1}{2}$"
Hex. nut, welded into position
Kieselguhr insulation
$\frac{3}{8}$" transite
Post
Post
Aluminum dial 6" diam.
Iron frame
1"
Flat pulley
Sprocket drive
Brass bearing
12"
Iron supports
Nº 22 gage galvanized sheet iron

Courtesy of U. S. Bureau of Mines

FIG. 1.—Davis Plastometer Retort and Furnace Assembly.

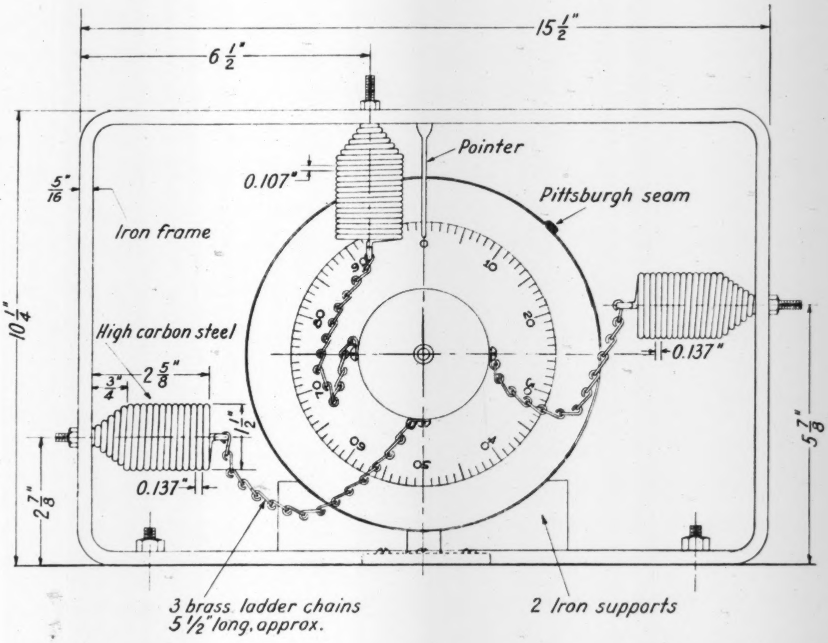
(*b*) *Furnace.*—A horizontal, electric-tube furnace, 12 in. in length, 8 in. in outside diameter, and 2 in. in inside diameter, as shown in Fig. 1, used for heating. The open ends of the furnace shall be equipped with transite covers which fit around the two extending axles. The clearance between the inside wall of the furnace and the outside wall of the retort shall be $\frac{1}{16}$ in.

(*c*) *Indicating Equipment.*—Equipment for indicating the torque developed by the heated coal as shown in Fig. 2. This shall consist of an aluminum dial with a 100-division graduated scale and shall be mounted on a flat pulley which is connected by three lengths of ladder chain to three springs, as shown in Fig. 2. There shall be provided two helical extension springs, slightly open wound so that there will be no stress on the springs when they are under "no load." They shall be made of the best quality spring steel. The dimensions of these springs shall be as follows:

Outside diameter of coil, in.	1.5
Diameter of wire, in.	0.137
Length of straight coil part, in.	1.875
Length of tapered coil part, in.	0.75

An additional spring shall be provided of the same dimensions, except that the diameter of the wire shall be 0.107 in.

One end of each spring shall have a "small eye ($\frac{5}{16}$ in. in inside diameter) over center;" the other end shall be tapered and provided with a swivel threaded bolt and nut. Dimensions of the three springs may be modified slightly to meet those of stock springs carried by individual manufacturers.


15 1/2"
6 1/2"
Pointer
0.107"
Pittsburgh seam
5/16"
Iron frame
10 1/4"
High carbon steel
3/4"
2 5/8"
1 1/2"
0.137"
0.137"
2 7/8"
5 7/8"
10
20
30
40
50
60
70
80
90
3 brass ladder chains
5 1/2" long, approx.
2 Iron supports

Courtesy of U. S. Bureau of Mines

FIG. 2.—Davis Plastometer Indicating Equipment.

Each spring shall be calibrated over its safe extension range in terms of kilogram-centimeters (or pound-inches) as expressed in dial divisions on the scale of the dial. This may be accomplished by attaching weights to a chain connected to the pulley in the same plane as that of the ladder chain joining the pulley to the individual spring being calibrated.

Sample

3. A representative gross sample of coal shall be collected and prepared in accordance with the Standard Method of Sampling Coal for Analysis (A.S.T.M. Designation: D 21) of the American Society for Testing Materials.[3] Approximately 15 lb. of coal crushed to pass a 4750-micron (No. 4) sieve shall constitute the laboratory sample.

Preparation of Sample

4. The 15-lb. laboratory sample shall be ground to pass an 840-micron (No. 20) sieve and shall be reduced by riffling to about 200 g. To prevent changes in plastic properties by oxidation, it is recommended that the coal shall not be dried other than by air-drying. The coal shall be tested for plastic properties as soon as possible after preparing the sample.

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[3] 1942 Book of A.S.T.M. Standards, Part III, p. 11.

Procedure

5. An 18-g. portion of the sample passing an 840-micron (No. 20) sieve shall be charged into the retort, which shall then be assembled by screwing in place the rear head containing the thermocouple well. The charged retort shall be placed in the furnace and the retort rotated continuously at a constant speed of 2 rpm. by means of an electric motor directly-connected to a reducing gear which in turn drives the retort through sprocket wheels connected by a chain. The charge shall be heated at a rate of 8.5 ± 0.2 C. per min. up to 300 C. and thereafter at 3.0 ± 0.1 C. per min. (Note). The coal offers negligible resistance to stirring by the rabble arms until fusion sets in. At this point the temperature and time shall be noted and the amount of resistance shall be read from the dial. Temperatures and resistances shall then be observed and recorded at intervals of 1 to 2 min. within the plastic temperature range. The 1-min. readings shall be taken when resistances are changing rapidly. Readings of temperatures and resistances shall be observed and recorded until the resistance again becomes zero.

NOTE.—Some low-volatile coals, certain high-volatile A coals, most high-volatile B and C coals, some blends containing major proportions of coals from these groups (Standard Specifications for Classification of Coals by Rank (A.S.T.M. Designation: D 388) of the American Society for Testing Materials[4]), and badly oxidized coals of all bituminous groups under the conditions of this test may show no resistance. In most instances, such coals may be made to fuse by employing a higher rate of heating.

Number of Tests

6. All tests shall be made in duplicate and the average values reported.

Calculation and Report

7. (*a*) *Calculation.*—The observed temperatures shall be corrected to the "true temperature" of the coal charge by adding experimentally predetermined corrections found from temperature measurements taken in the center of the retort to those temperatures observed regularly in the thermocouple well during the course of the test. This correction amounts to 10 C. in the retort with the procedure described in this method.

(*b*) *Report.*—The report shall include the following:

(*1*) The fusion temperature of the coal corresponding to the first appreciable resistance to stirring as observed from the movement of the dial,

(*2*) The temperature of maximum fluidity or "minimum resistance," after a more or less complete initial fusion of the coal, when the observed resistance reaches a minimum. This region of maximum fluidity may extend over a short temperature range, in which instance the temperature range corresponding to the constant maximum fluidity or "minimum resistance" may be reported,

(*3*) The temperature of solidification when the maximum resistance is shown,

(*4*) The temperature of the end of the plastic range after the semi-coke has been broken up by the stirring and no further resistance is observed, and

(*5*) A chart in which kilogram-centimeters (or pound-inches) are plotted as ordinates against "true" temperatures as abscissas may be used to summarize the results of the test.

Reproducibility

8. All characteristic temperature points should agree within 5 C. Resistance values at the temperature or temperature range of maximum fluidity and at the temperature of solidification should agree within 20 per cent.

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[4] 1942 Book of A.S.T.M. Standards, Part III, p. 1.

APPENDIX III

PROPOSED METHOD OF TEST FOR PLASTIC PROPERTIES OF COAL BY THE GIESELER TYPE PLASTOMETER[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test is intended to determine the relative plastic behavior of coal when heated under prescribed conditions in the absence of air.[2] The test is useful in predicting the coking behavior of bituminous coals and blends and for determining the extent of weathering of coal when results for the fresh coal are known.

Apparatus

2. A convenient form of the Gieseler plastometer is shown schematically in Fig. 1. The apparatus shall consist of the following:

(*a*) *Retort.*—A steel retort consisting of five pieces as shown in Fig. 2. The retort base or crucible shall be cylindrical, $\frac{27}{32}$ in. in inside diameter and $\frac{5}{8}$ in. in depth, with exterior threads for joining the crucible to the shaft or barrel. The outside diameter of the threaded part of the base shall be 1 in. and the dimensions of the remaining lower part of the base shall be optional, but designed to fit into the loading device, Fig. 3. The main length of the barrel shall be $\frac{3}{8}$ in. in inside diameter. The lower end of the barrel shall be $\frac{27}{32}$ in. in inside diameter to a height of $\frac{1}{2}$ in. above the top of the threaded section. The top end of the barrel shall be $\frac{5}{8}$ in. in inside diameter to a depth sufficient to allow clearance for the axle of the plastometer head when the apparatus is assembled. A gas-exit tube shall be located on the barrel at a position convenient to carry off the volatile products out of the retort during a test. A steel washer $\frac{1}{16}$ in. in thickness shall be provided to fit between the lower end of the barrel and the top of the crucible when the retort is assembled. This washer shall have an opening $\frac{3}{8}$ in. in diameter in its center. The stirring shaft of the retort shall be constructed of $\frac{5}{32}$-in. drill rod and equipped with four rabble arms. The lower end of the stirrer shall be tapered to a point to fit into the pivotal depression in the crucible. The rabble arms shall be so arranged that the lowest arm will be $\frac{1}{16}$ in. from the crucible bottom when the retort is assembled. The rabble arms shall be $\frac{1}{16}$ in. in diameter and $\frac{1}{4}$ in. in length and shall be placed at 90-deg. intervals around the stirrer and $\frac{1}{8}$ in. apart, center to center. The upper end of the stirrer shall be cut to fit into the receiving slot on the lower end of the axle in the plastometer head. A sleeve guide shall fit into the upper part of the barrel to act as a second bearing for the stirrer as shown in Fig. 2. The sleeve guide shall fit closely in the barrel and have an inside diameter large enough to permit free movement of the stirrer.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke. Published as information, June, 1943.

[2] For information concerning the experimental work on which this method is based, the following references may be consulted:

R. E. Brewer and J. E. Triff, "Measurement of Plastic Properties of Bituminous Coals. Comparison of Gieseler and Davis Plastometer and Agde-Damm Dilatometer Methods," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 11, p. 242 (1939).

K. Gieseler, "Messung der plastischen Eigenschaften erhitzter Kohlen," *Glückauf*, Vol. 70, p. 178 (1934).

H. Macura, "Neue Erkenntnisse über das Verhalten von Steinkohlen bei der Erhitzung. I Teil: Zur Kenntnis des Erweichungsvorganges," *Oel u. Kohle ver. mit Erdoel u Teer*, Vol. 14, p. 1097 (1938).

K. Hoehne, "Untersuchungen über die Bildsamkeit von Steinkohlen," *Glückauf*, Vol. 75, p. 941 (1939).

G. Jung, "Auswirkungen der Alterung auf die Plastizität von Kokskohlen," *Glückauf*, Vol. 71, p. 1141 (1935).

G. C. Soth, and C. C. Russell, "The Gieseler Method for Measurement of the Plastic Characteristics of Coking Coals," see p. 1176.

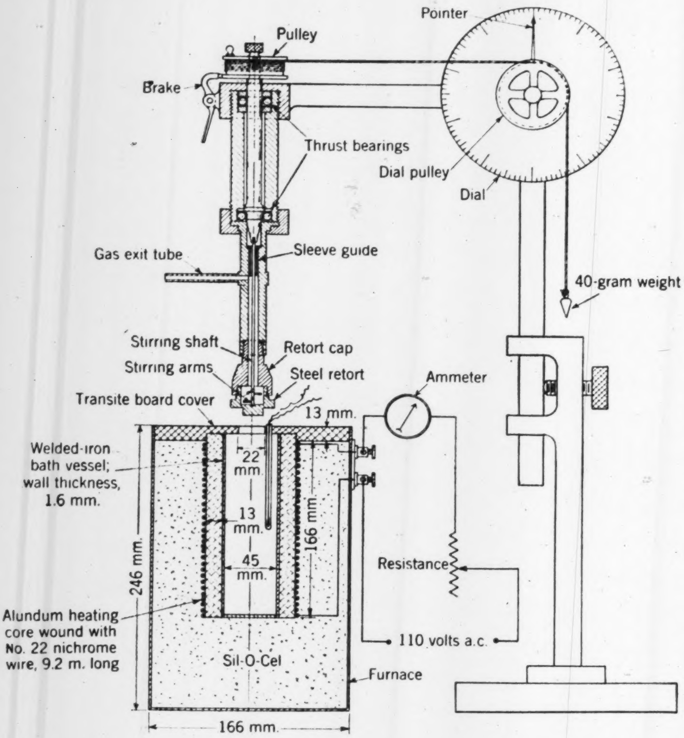
Pointer
Pulley
Brake
Thrust bearings
Dial pulley
Dial
Gas exit tube
Sleeve guide
40-gram weight
Stirring shaft
Retort cap
Stirring arms
Steel retort
Ammeter
Transite board cover
13 mm.
Welded-iron bath vessel; wall thickness, 1.6 mm.
22 mm.
13 mm.
166 mm.
45 mm.
246 mm.
Resistance
Alundum heating core wound with No. 22 nichrome wire, 9.2 m. long
110 volts a.c.
Sil-O-Cel
Furnace
166 mm.

FIG. 1.—Gieseler Type Plastometer.

(*b*) *Plastometer Head.*—The plastometer head shall contain an axle mounted in ball bearing races. The lower end of the axle shall be slotted to connect with the stirrer. The upper end of the axle shall be equipped with a pulley which fits loosely enough to revolve without turning the axle. The upper end of the axle shall be provided with a milled-headed screw to tighten the pulley to the axle. The plastometer head shall be equipped with a brake to prevent movement of the pulley. A cord shall be wound around the pulley and shall be passed over a second, vertically mounted pulley of the same outside diameter. The second pulley shall have a pointer to indicate movement around a dial which shall be divided into 100 divisions for 360 deg. The end of the cord passing over the second pulley shall be loaded with a total weight of 40 g.

(*c*) *Furnace.*—The furnace for heating the retort shall be of such construction that a heating rate of 3 ± 0.1 C. per min. can be maintained up to at least 550 C.

The furnace shall contain a suitable metal or alloy bath, melting below 275 C. and of such composition to prevent overheating in spots. Among others, a molten solder bath (50 per cent lead, 50 per cent tin) with a thin layer of powdered graphite over the bath surface to minimize oxidation has proved satisfactory. Temperatures in the bath shall be measured with a thermocouple in a steel protecting tube. The protected couple shall be inserted into the bath so that the protecting tube touches the outside wall of the retort and the hot junction of the couple is at the same height as the center of the coal charge.

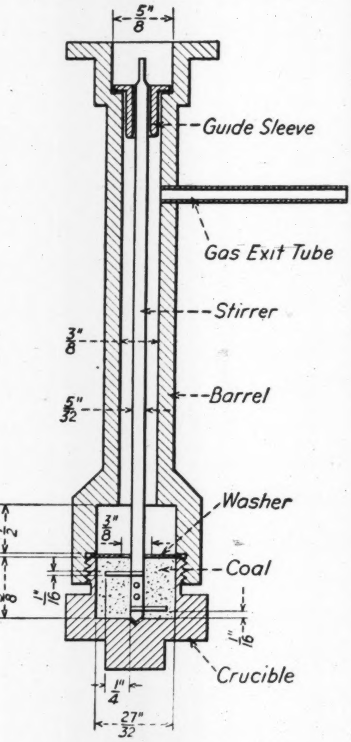

FIG. 2.—Gieseler Retort Assembly.

(*d*) *Loading Device.*—The loading device shall be designed so the coal may be uniformly packed in the retort under a total packing load of 10 kg. The arrangement is shown in Fig. 3.

Sample

3. A representative gross sample of coal shall be collected and prepared in accordance with the Standard Method of Sampling Coal for Analysis (A.S.T.M. Designation: D 21) of the American Society for Testing Materials.[3] Approximately 15 lb. of coal crushed to pass a 4760-micron (No. 4) sieve shall constitute the laboratory sample.

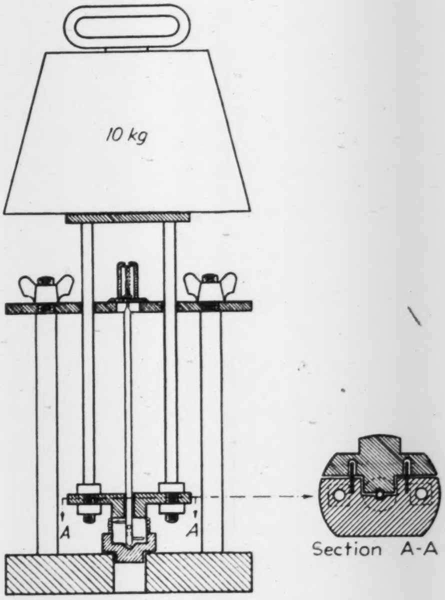

FIG. 3.—Loading Device.

Preparation of Sample

4. The 15-lb. laboratory sample shall be ground to pass an 840-micron (No. 20) sieve and shall be reduced by riffling to about 200 g. This 200-g. portion shall be quartered and one quarter shall be stage-crushed to pass a 500-micron (No. 35) sieve in such manner as to minimize production of fines. To prevent changes in plastic properties by oxidation, it is recommended that the coal shall not be dried other than air-drying at 10 to 15 C.

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[3] 1942 Book of A.S.T.M. Standards, Part III, p. 11.

above room temperature until the moisture loss is less than 0.1 per cent per hr. The coal shall be tested for plastic properties as soon as possible after preparing the sample passing the 500-micron (No. 35) sieve.

Procedure

5. (*a*) The open crucible with stirring shaft in place shall be mounted in the loading device as shown in Fig. 3. The prepared coal passing the 500-micron (No. 35) sieve shall be charged into the crucible by alternately adding the coal in small increments and packing by lowering the cage of the loading device until the coal charge is level with the top of the crucible. Tapping the crucible during packing shall be avoided to prevent size segregation. The charge shall then be compressed for 15 min. under the 10-kg. load. This manner of charging gives a bulk density of approximately 0.87 g. per cu. cm. or 54.3 lb. per cu. ft.

(*b*) The loading device shall be removed from the charged crucible without disturbing the position of the stirrer embedded in the coal charge. The washer shall be placed on top of the crucible and the barrel shall be screwed on the crucible and tightened. The guide sleeve shall then be inserted in the top end of the barrel and the barrel screwed on the plastometer head making sure that the top end of the stirrer is in the slot in the lower end of the axle in the head. The assembled apparatus shall be lowered until the retort is immersed to a depth of 3 in. in the molten metal bath contained in the furnace previously heated to 300 C. The furnace shall then be covered with the transite board top. The heating control and heat capacity of the bath shall be such that the retort will attain a temperature of 300 C. in 10 min. after insertion into the heated bath. Thereafter, the retort shall be heated at a rate of 3 ± 0.1 C. per min.

(*c*) As soon as the retort has reached 300 C. the brake on the pulley shall be released and the pointer set at zero. At the first detectable continuous movement of the pointer the corresponding temperature shall be recorded as the "initial softening temperature." Thereafter, readings of time, temperature, and pointer movement shall be taken at 1-min. intervals. Readings may be taken less often than each minute if a longer interval gives a sufficient number of points for a satisfactory test. If the movement of the pointer becomes greater then five dial divisions per minute, the brake shall be applied and it shall be released only just preceding and during readings. In such instances, the pointer movement shall first become uniform and its rate shall be timed with a stop watch. These periodic readings shall be made until the pointer movement is again less than five dial division per minute, at which time the use of the brake shall be discontinued. If it is found necessary during the test to rewind the cord on the pulley on the plastometer head, the screw which tightens the pulley to the axle shall be loosened so that, in the rewinding, the stirrer in the coal sample is not disturbed. The test shall be continued until pointer movement is no longer evident.

Number of Tests

6. All tests shall be made in duplicate and the average values reported.

Report

7. From the observed times and dial readings the corresponding movement of the pointer shall be calculated in dial divisions per minute. Bituminous coals show a wide range of plasticity or fluidity. For this reason it is convenient to plot dial divisions per minute as ordinates on a logarithmic scale against temperatures as abscissas on a uniform

arithmetical scale. Characteristic temperature points to be noted especially shall be:

(*1*) The initial softening temperature corresponding to the first detectable continuous movement of the pointer,

(*2*) The fusion temperature corresponding to five dial divisions per minute on the ascending portion of the curve,

(*3*) The temperature of maximum fluidity corresponding to the maximum rate of pointer movement, and

(*4*) The solidification temperature corresponding to the temperature at which the pointer shows no further movement.

Reproducibility

8. All characteristic temperature points for duplicate tests should agree within 5 C. Maximum rate of pointer movement at the maximum fluidity temperature should agree within 20 per cent.

APPENDIX IV

PROPOSED METHOD OF TEST FOR CARBONIZATION PRESSURE OF BITUMINOUS COAL[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method[2] is a small-scale laboratory test intended for determining the pressure developed by coal during carbonization.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Oven.*—An oven built in two halves in such a manner as to reduce the influence of friction; one half of the furnace shall be in a fixed position, while the other shall be mounted on four ball-bearing wheels which run on angle iron tracks perpendicular to the oven walls. The carbonization shall be accomplished in a rectangular chamber built of silica brick which shall be heated on opposite sides by a burning gas-air mixture. The coking and combustion chambers shall be insulated by lightweight fire brick, and shall be reinforced by an angle-iron frame. The width of the coking chamber may be varied either by original design or by appropriate inserts. The floor of the chamber shall be lined with ¼-in. silica brick to prevent excessive wear and all other refractory surfaces that come in contact with the coal charge shall either be coated with silica cement or lined with silica brick. A cross-section of the oven is shown in Fig. 1.

(*b*) *Combustion Unit.*—Uniform heating over the whole face of the chamber shall be accomplished by special burners of a simplified type. Two burners, each of which consists of two concentric pipes, shall be assembled into a unit by screwing the ends of the gas inlets into a tee. The air inlets shall be arranged in a similar manner. Details of the construction are shown in Fig. 2. The units shall be fastened in place with four small pieces of strap iron which shall be bolted to the frame. The ends of the burners shall project about ½ in. into the burner ports and the open space around the burners shall be closed with packing of wet asbestos paper or cement.

The two identical surface combustion units shall be narrow chambers 8½ by 8½ by 1 in. filled with ¼ by ½-in. silicon carbide crystals. The silicon carbide, being a good conductor with a large heat capacity, tends to make the temperature of the hot walls more uniform, and its mechanical strength reinforces these walls. The units may be held at any selected temperature between 400 and 1300 ± 10 C. by means of a heat control device. The temperature shall be regulated by means of a platinum platinum-rhodium thermocouple balanced in a potentiometer circuit. A galvanometer shall be used in this circuit to operate the photoelectric relay which controls the solenoid valves in the gas and air lines.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke. Published as information, June, 1943.

[2] For information concerning the experimental work on which this method is based, see the paper by W. Fuchs, A. G. Sandholt, J. A. Taylor, and A. W. Gauger, "Studies Concerning the Pressure Developed During the Carbonization of Coal," Pennsylvania State College, Mineral Industries Experiment Station, ***Bulletin 34*** (1941).

(*c*) *Measuring and Recording Apparatus.*—A triple-layer stainless steel bellows $1\frac{1}{2}$ in. in diameter, as shown in Fig. 3, capable of withstanding a maximum pressure of 600 psi. The pressure shall be transmitted to the bellows from the furnace through an adjustable brass tube and measured on a Bourdon type gage or on an automatic recorder. The system shall be filled with a suitable liquid, either water or oil. The movement of the wall is usually of the order of $\frac{1}{32}$ in. or less.

Sample

3. A representative gross sample of coal shall be collected and prepared in accordance with the Standard Method of Sampling Coal for Analysis (A.S.T.M. Designation: D 21).[3] A sample of 2300 g. shall be used in the 3-in. oven and 3200 g. in the $4\frac{1}{2}$-in. oven.

Procedure

4. (*a*) After lighting the burners at the pilot holes, the oven shall be slowly brought to the specified temperature; this usually takes a minimum time of 3 to 4 hr.

(*b*) The oven temperature shall be regulated to 1100 C.

(*c*) The steel bellows shall be set so that the end protrudes $\frac{1}{16}$ in. from the plate. If excessive movement occurs during a test, the bellows shall be heated to remove any air or gas.

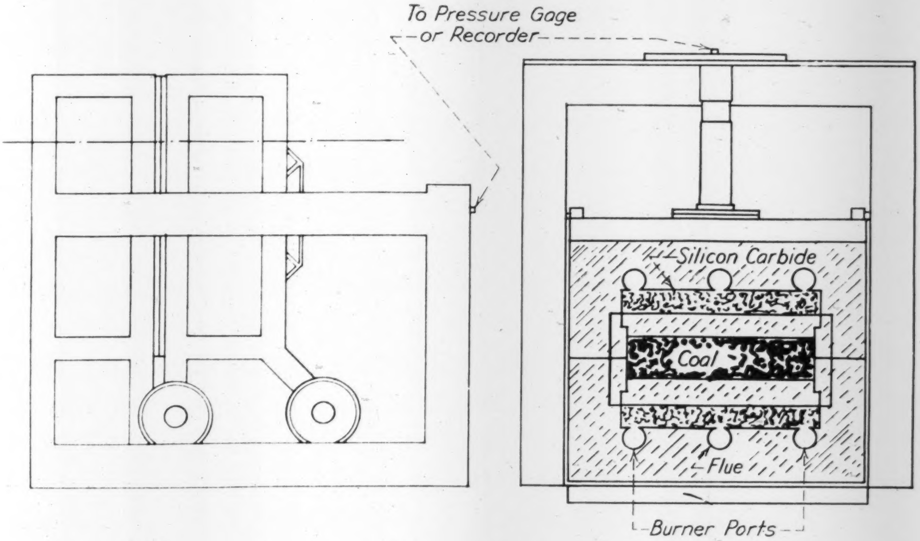

FIG. 1.—Oven.

(*d*) The pressure connecting rod shall be put in place and the gage set to read 15 psi. This setting shall be made by hand rotation of the rod.

(*e*) The sample shall be weighed and mixed, its bulk density determined and then transferred to the charging hopper (2300 g. in the 3-in. oven and 3200 g. in the $4\frac{1}{2}$-in. oven).

(*f*) Charging shall be made according to a time schedule; the top brick shall be removed 10 sec. before the minute, the hopper shall be placed on top of the oven directly over the chamber, and the charge dropped.

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[3] 1942 Book of A.S.T.M. Standards, Part III, p. 11.

(*g*) The headpiece shall be placed over the oven, the fume hood closed, and any insulation or protection for the hood shall be put in place. The headpiece of two whole bricks which cover the top of the chamber shall be placed on top during the test. These two bricks shall be grooved so that when they are placed together a small rectangular slot $\frac{1}{2}$ by $1\frac{1}{2}$ in. is formed for the escape of volatile matter. The purpose of the headpiece is to make conditions similar to those in actual practice and to permit combustion of volatile matter without a direct flame coming in contact with the charge.

(*h*) If there is no provision for automatic recording, the pressures shall be noted at regular intervals.

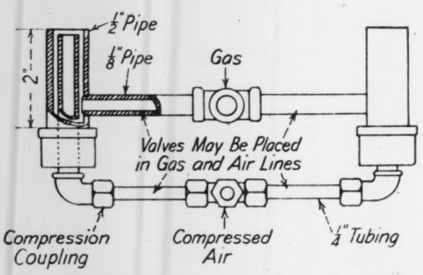

FIG. 2.—Burners for Combustion Unit.

(*i*) Any pressure peaks and minimums that do not occur at the normal time for a reading shall be recorded. The data may be plotted as they are taken if automatic recording is not available.

(*j*) After the final peak has been recorded and the pressure has reached a steady value, the headpiece shall be removed and the volume of the coke shall be determined. If it is desirable to know volume change occurring during coking process, an estimate may be obtained by filling the carbonization chamber after completion of the test with coke breeze sized between 3 and 6 mesh. From the volume of the coke breeze, the change in volume accompanying the change of coal to coke can be determined. A reliable indication that coking is completed is that the burning volatile matter changes from a smoky, yellow flame to one almost colorless.

(*k*) If the coke is to be studied further, the charge shall be retained in the oven for 5 min. after the smoky flame has disappeared. If the coke is to be discarded, it may be discharged as soon as the top volume has been determined.

(*l*) To discharge the oven, the air and gas shall be turned off, the service lines between the two sections disconnected, a suitable pan containing about $\frac{1}{16}$ in. of water placed under the oven, the pressure rod removed, the oven opened, and the coke raked out.

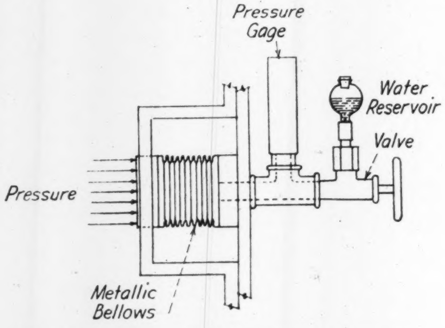

FIG. 3.—Bellows and Pressure Recording Apparatus.

(*m*) The oven shall be prepared for another test by pushing the oven sections in place, connecting and adjusting the gas and air lines, placing the insulating brick over the oven chamber, and returning the pressure rod to its proper position.

Calculation

5. The carbonization pressure shall be calculated in pounds per square inch of oven wall.

Reproducibility

6. In order to obtain a critical estimate of the accuracy of pressure data obtained by this method with one sample in a

particular test under fixed conditions, it is necessary to know the theoretical maximum pressure to be expected with the sample under consideration, or at least the actual maximum pressure under the circumstances. At present, theoretical maximum pressures cannot be calculated, and actual maximum pressures can be found only by a statistical analysis of a large number of data, for example, 100 measurements with the same coal. Because of these conditions, the only criteria now available for judging the merits of a given method of measurement are the comparison with empirical standards and the duplicability of numerical values. Duplicate tests in a 4½-in. oven may be expected to check better than plus or minus 10 per cent when rigid control in preparing the sample and operation of the oven is exercised.

APPENDIX V

PROPOSED METHOD FOR MEASUREMENT OF PRESSURES DEVELOPED DURING CARBONIZATION OF COAL BY THE MOVABLE WALL OVEN[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method[2] of test is intended for determining the pressure developed by coal during carbonization for the purpose of obtaining information for the evaluation of the safety of coals for use in by-product coke ovens.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Test Oven.*—A small coke oven, as shown in Figs. 1 and 2, heated in such a manner that coking takes place at a rate and in a manner substantially similar to commercial ovens. The oven chamber shall be 12 in. in width, 42 in. in height, and 28 in. in length, capable of holding a sample of approximately 400 to 450 lb. of coal. Both of the side walls shall be heated by gas, controlled manually with needle valves. Each heating wall shall have multiple flues with offsets in alternate tiers to produce turbulent flow of the heating gases. One wall shall be mounted on a carriage arranged to move freely on supporting rails and casters; the other shall be fixed and rigid. The movable wall shall be connected to the lever system and suitably counterbalanced or otherwise arranged so that pressures exerted on the movable wall shall be transmitted to the measuring device without undue loss due to friction or other causes.

(*b*) *Oven Walls.*—The oven walls shall be constructed of brick satisfactory to withstand the pressures developed at the test temperatures without bending or distortion. The brickwork exposed to high temperatures shall be of the low thermal expansion type. A good grade of insulating brick should be used to minimize heat losses. The outer course shall be of No. 1 fireclay brick. The stationary wall shall be 33 tiers high at $2\frac{5}{8}$ in. each. The movable wall shall be 29 tiers high and mounted on the carriage $10\frac{1}{2}$ in. above the floor. Openings shall be provided for measuring the temperature of the heating flues by means of an optical pyrometer. The oven shall have one door, the opposite end being closed and containing a carborundum block having 13 holes each $\frac{5}{16}$ in. in diameter spaced 1 in. apart for the insertion of thermocouple wells. The back end of the oven shall have an opening 37 in. above the oven floor for the purpose of levelling the top of the charge by means of a small rake.

(*c*) *Structural Supports.*—The oven wall brickwork and levers shall be bound by 8 in. channels and $\frac{7}{8}$ in. tie rods in such a manner that there is no undue loss in the pressures that are to be measured.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke. Published as information, June, 1943.

[2] For information concerning the experimental work on which this method is based, see the paper by Charles C. Russell, "Measurement of Pressures Developed During the Carbonization of Coal," Am. Inst. Mining and Metallurgical Engrs., *Technical Publication No. 1118*, p. 113 (1939).

(*d*) *Lever System.*—A lever system, as shown in Fig. 1, connected between the movable wall of the oven and a weight on a platform scale. The lever ratio shall be 7 to 1. About 1300 lb. of pig lead shall be provided in a tray connected to the lever system and resting on the platform scale.

(*e*) *Charging Pipe.*—A charging pipe, which also serves as an outlet for the volatile products, provided at the top of the oven. A bottom-discharging hopper shall be provided that will hold the entire sample of coal to be tested so that the sample may be charged into the oven through the top in one continuous stream.

(*f*) *Auxiliary Apparatus.*—For removal of the oven door, a pantograph type lever supported by a rod suspended from an overhead trolley running on the bottom flange of an I-beam as shown in Fig. 2. A long steel rake as well as a sheet metal chute shall be provided to facilitate removal of coke from the oven.

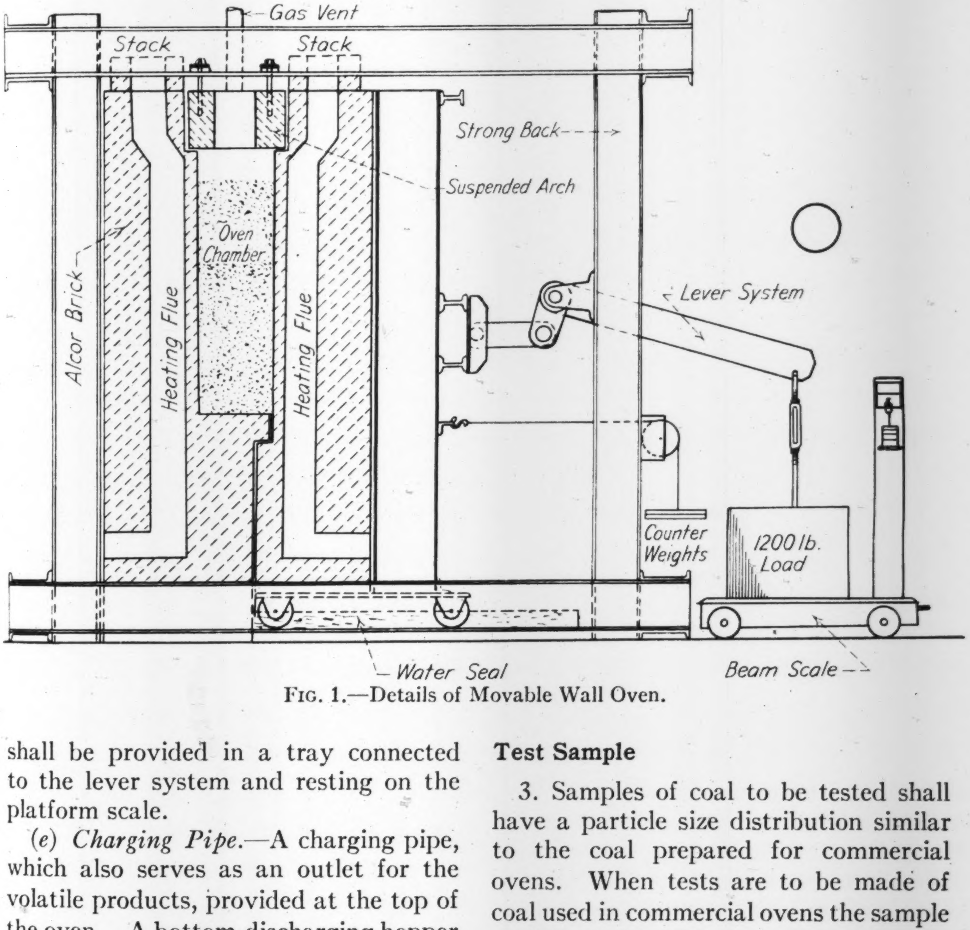

FIG. 1.—Details of Movable Wall Oven.

Test Sample

3. Samples of coal to be tested shall have a particle size distribution similar to the coal prepared for commercial ovens. When tests are to be made of coal used in commercial ovens the sample shall be obtained in accordance with the Standard Method of Sampling Coal for Analysis (A.S.T.M. Designation: D 21) of the American Society for Testing Materials.[3] When it is impossible to obtain a

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[3] 1942 Book of A.S.T.M. Standards, Part III, p. 11.

bulk density of the sample in the oven as high as the maximum bulk density in commercial ovens, it shall be permissible to air dry the sample before charging until the surface moisture of the coal has evaporated. Drying of coal by application of heat is not permissible.

Procedure

4. (*a*) Before the sample of coal is placed in the oven, the oven shall be heated so that the temperature in the empty oven chamber is 1800 F.

(*b*) The volume of the oven shall be determined by measurement of the oven while hot.

(*c*) The test sample shall be weighed carefully before being placed in the oven. At least 5 per cent more coal shall be placed in the oven than the normal weight of the charge in order to supply sufficient excess for leveling of the charge.

(*d*) After the sample is charged into the oven, it shall be carefully levelled so that the top of the charge is of uniform height and level with the bottom of the levelling door.

(*e*) The pressure measuring device shall be adjusted to zero or otherwise adjusted so that measurement of pressures can be obtained from the start of the test.

(*f*) Immediately after the charge has been levelled, the first pressure reading shall be obtained. Thermocouple wells shall be inserted into the test sample as soon after charging as possible. The weight of coal withdrawn from the oven by levelling shall be determined and the net weight of the test sample calculated. The bulk density of the test sample shall be calculated from the weight of the charge and the volume of the oven to the top of the test sample.

(*g*) Pressure measurements shall be made every 30 min. for the first 2 hr. and every hour thereafter until the center

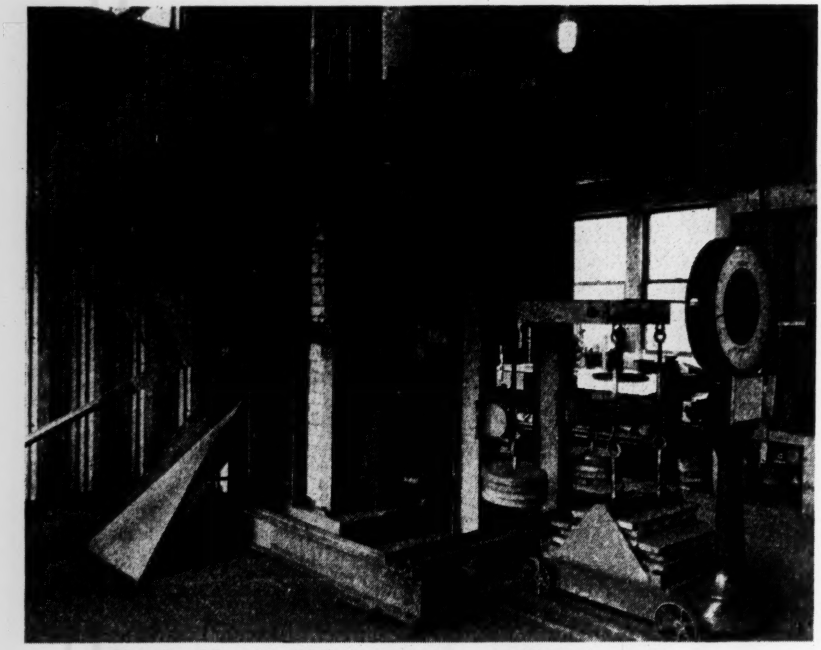

FIG. 2.—Movable Wall Oven Showing Auxiliary Apparatus.

thermocouples indicate a temperature of 390 F. Careful attention to pressure changes shall be made and significant changes in pressure shall be recorded until the center thermocouple indicates a temperature of 1110 F.

(*h*) Temperatures of the charge in the oven shall be recorded every hour throughout the duration of the carbonizing period.

(*i*) Temperatures of the heating walls or flues shall be maintained at 2500 F. and shall be recorded at intervals throughout the carbonizing period to indicate the constancy of the heat being applied to the test sample. The temperature of the flues shall not vary more than 50 F. during the test.

(*j*) After the temperature at the center of the test sample has reached 1110 F. and it has been found that there are no further significant changes in pressure, the coke may be withdrawn from the oven.

Report

5. The data obtained from each test shall be recorded graphically. The pressures developed during carbonization, expressed in pounds per square inch, shall be plotted against time in hours after charging. Other significant data such as the bulk density of the test sample, pulverization of the test sample, the name of the coal or mixture tested, the average flue temperature, and date of test shall be recorded on the same sheets.

Reproducibility

6. Provided the flue temperatures are approximately 2500 F. and the initial bulk densities of samples used in duplicate tests are within 0.5 lb. per cu. ft. of one another, the reproducibility of pressure measurement by this method should be within 0.3 psi.

NOTE.—It is impossible at present to convert by calculation the results of tests obtained on coal of one bulk density with those of another bulk density. In order to duplicate results, two tests must be made at substantially the same bulk density.

APPENDIX VI

PROPOSED METHOD OF TEST FOR EXPANSION PROPERTIES OF COAL FOR USE IN BY-PRODUCT COKE OVENS BY MEANS OF THE BETHLEHEM TEST OVEN[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method[2] of test covers the procedure for determining quantitatively the expansion (or contraction) behavior of coking coals and their mixtures in the Bethlehem Expansion Tester for the purpose of obtaining information for preventing or reducing progressive damage to the brick linings of coke ovens.

NOTE.—The coke obtained from this test may be employed for limited determination of physical and chemical properties since it has been prepared under conditions relatively comparable to commercial oven practice.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Test Oven.*—A test oven consisting of a rectangular carbonizing chamber having an area of 300 sq. in., heated unidirectionally by a combustion chamber under the floor, and fitted with a weighted rectangular piston equipped with indicating or recording attachments for measuring piston displacement.

(*b*) *Carbonizing Chamber.*—A carbonizing chamber 10 to 11 in. in width by 27 to 30½ in. in length (dimensions when cold) and of sufficient depth to contain a 35 to 45 lb. sample of pulverized coal 3¾ to 4 in. in depth. The floor of the carbonizing chamber shall be of first quality silica brick 2½ in. in thickness and the sides of either silica or fire clay brick. Two opposite sides shall be removable for discharging the coke after completion of the test.

(*c*) *Floor Heating Arrangement.*—Three upwardly communicating horizontal passages, the first and lower of which shall accommodate a simple bunsen-type gas jet burner with the third or topmost under the carbonizing chamber and leading off to a suitable stack or chimney. The combustion passage design shall be such that the entire oven floor is heated uniformly, a minimum of 90 per cent being within plus or minus 45 F. of the average floor temperature. Effective ceramic thermal insulation shall be provided about the oven section and combustion section. Properly braced and stayed steel framework shall maintain rigidity of the oven under test and temperature stresses.

(*d*) *Piston.*—A piston conforming to the shape of the carbonizing chamber with a wall clearance of ⅛ in. and maintained by guides or rollers. It shall consist of a metal frame with plane sheet steel face, lined with insulating brick and fitted with a number of vent holes each ½ in. in diameter to carry off vapors evolved during the test. Its weight shall be such that a pressure of 2.25 psi. bears upon the coal charge.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-5 on Coal and Coke. Published as information, June, 1943.

[2] For information concerning the experimental work on which this method is based, see the paper by W. T. Brown, "Coal Expansion," *Proceedings*, Am. Gas Assn., Vol. 20, 1938, p. 640; also H. S. Auvil and J. D. Davis, "Determination of the Swelling Properties of Coal During the Coking Process," U. S. Bureau of Mines, *Report of Investigations No. 3403*, p. 3, (1938).

(*e*) *Dial Gages.*—For measuring the movement of the piston, a pair of accurate dial gages or a similar arrangement connecting the piston and the steel oven framework. Alternatively, a reference pin and arm may be used to indicate the piston movement to within 0.03 in.

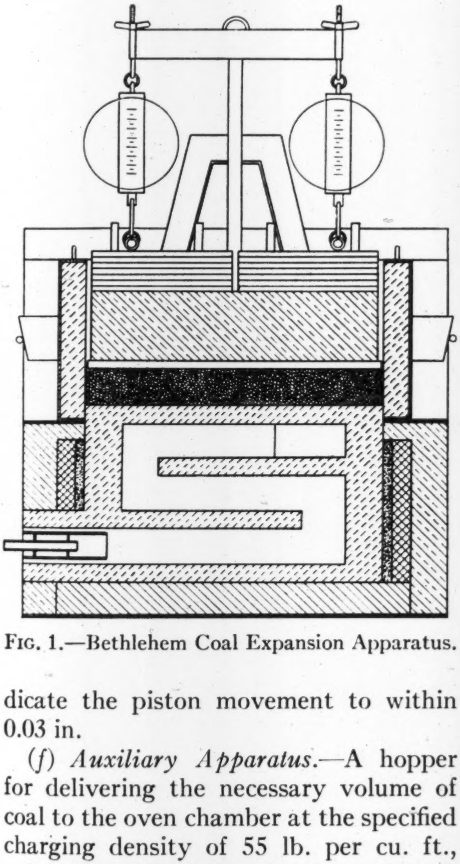

FIG. 1.—Bethlehem Coal Expansion Apparatus.

(*f*) *Auxiliary Apparatus.*—A hopper for delivering the necessary volume of coal to the oven chamber at the specified charging density of 55 lb. per cu. ft., a levelling device for the charged coal, a piece of ¼-in. mesh wire screen to be placed between the coal surface and the piston to prevent sticking, a pyrometric temperature measuring device for the floor of the carbonizing chamber, properly protected for insertion in the upper combustion passage, a cover for the chamber when being heated between tests, and tools for handling the removable oven sides and for removing the coke following the tests.

Test Sample

3. (*a*) *Test Sample.*—The test sample shall consist of coal or coal mixtures fully prepared as for use in a commercial coke oven. The sample shall be reduced to approximately 45 lb. in accordance with the Standard Method of Sampling Coal for Analysis (A.S.T.M. Designation: D 21) of the American Society for Testing Materials.[3]

(*b*) *Comparative Sample.*—For tests to determine comparison with coke oven operation, the sample shall consist of coal or mixtures of coals which have been crushed to a fineness of 85 to 90 per cent passing a 3360-micron (No. 6) sieve and reduced to approximately 45 lb. in accordance with Standard Method D 21.[3] The air-dried sample shall contain about 2 per cent moisture.

Oven Heating

4. The temperature of the lower side of the carbonizing chamber floor in the upper passage or flue shall be maintained at 2350 ± 25 F. during the test period. Temperature readings shall be taken every 30 min. for control which shall be effected by gas and air adjustment and by manipulation of a stack damper. An interval of 6 to 8 hr. between tests shall be observed for readjustment and equalization of the floor temperatures. For tests to determine comparison with coke oven operation, a flue temperature 100 F. below that of the large ovens may be maintained. Automatic control is desirable.

NOTE.—To prevent damage to the silica brick in the oven, it is necessary to exercise extreme care in bringing a new oven up to test temperature and the oven should be maintained above 1000 F. between periods of activity. Rapid temperature passage through the silica transition range must be avoided.

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[3] 1942 Book of A.S.T.M. Standards, Part III, p. 11.

Procedure

5. (*a*) Approximately 35 to 45 lb. of the properly prepared sample shall be carefully weighed into the charging hopper. The exact weight shall be calculated from the depth of the coal charge, which shall be uniform for all tests, from the hot oven dimensions, and from the specified bulk density of the test sample of 55 lb. per cu. ft. The hopper shall be placed over the chamber after removing the cover and the sample then dropped into the oven which has been maintained at the test temperature for 6 to 8 hr. previous. The sample shall then be uniformly levelled to the prescribed depth. The $\frac{1}{4}$-in. mesh wire screen and the piston shall then be placed upon the coal and the latter connected to the dial gages or other displacement measuring devices. These shall be set at some intermediate point to provide for possible contraction. Door-luting-mud shall be used to seal the joints of the removable sides of the oven to prevent ingress of air and burning of the charge. The flue temperature and readings of the piston movement shall be taken every 30 min. during the test period.

(*b*) The test shall be considered completed when the sample has coked through and has become rigid, ending piston movement. This may be within 6 to 9 hr. At this time the gages shall be disconnected and the piston removed. The removable sides of the oven shall be removed and the coke pushed from the carbonizing chamber and quenched with water. After brushing out the carbonizing chamber, the sides and cover of the oven shall be replaced for the preheating period of the next test.

NOTE.—The coke may be dried and weighed for yield and then subjected to other tests as desired.

Report

6. The averages of the two sets of gage readings obtained shall be converted to percentage expansion or contraction in reference to the initial depth of the test sample. Results may be plotted against time upon a suitable chart. Final expansion or contraction shall be reported to the nearest 1 percent and for a specified bulk density if other than 55 lb. per cu. ft.

Reproducibility

7. The percentage expansion or contraction obtained in duplicate tests should not differ by more than 2.

DISCUSSION

PRESSURES, STRAINS, AND OTHER PROPERTIES DEVELOPED DURING CARBONIZATION OF COAL

MR. V. J. ALTIERI[1] (*presented in written form*).—Appended to the Report of Committee D-5 are a number of methods for determining the expansion and pressure characteristics developed during the carbonization of coal. Application of one or another of these methods should furnish useful information concerning the properties throughout the coking period. It is believed, however, that more consideration should be given to certain factors that often become important in connection with specific problems in the field of coal carbonization; that while small-scale tests are needed to supplement and sometime replace the large or full-scale tests, much greater care is required in selecting and using small-scale tests; and, that the material being coked, for the purposes of the test, may be classified as a structural engineering material and handled by the conventional methods.

The object of this discussion is to outline the development and use of a small-scale engineering model of a coke-oven utilizing these ideas for the purposes listed in Section 1 (*a*) of the attached procedure. The procedure was written up in A.S.T.M. form in order to facilitate comparison with the corresponding methods appended to the Report of Committee D-5. It is hoped that making it thus available will result in greater use and better evaluation of the essential principles.

The consensus of opinion is that full-scale oven tests are the only accurate and thoroughly reliable means of ascertaining the coking properties of coal under the conditions met with in industrial use. In such tests, however, in order to determine the optimum conditions, it is usually necessary to run a number of tests while varying the blending and conditioning of the raw material, and also the full-scale operating conditions. Because of this situation, and because of the man power, time, and expense involved, it is often necessary to make preliminary tests, the results of which can be used as a basis in selecting the raw materials and carbonizing conditions for the full-scale tests. Moreover, situations often arise where only the small-scale test can be used; for instance, where it is required to reproduce conditions met with in different parts of a coke oven in order to determine localized stresses that may cause deferred damage; or, where there is not enough sample for making a full scale test. Under some conditions, therefore, only the small-scale test can be used; under other conditions, it often is advisable to run both the small-scale and the full-scale tests. There is a gigantic investment in by-product coke oven plants, and in the face of a decreasing supply of virgin coking coals, a large proportion of that investment may be impaired by the use, unwittingly, of coals or coal blends, or carbonizing conditions which insidiously exert power to cause what may be called deferred damage; namely, damage to ovens which becomes evident only after there has been a certain lapse of time. Whether it is immediate or deferred, the damage is a matter concerning the properties of materials existing within the coke ovens during the coking period. To keep abreast of developments in this materials field, therefore, it is necessary to use relatively accurate and reliable small-scale tests to supplement and sometimes to replace the large or full-scale tests.

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[1] Chief Chemist, Eastern Gas and Fuel Associates, Everett, Mass.

A glance at developments in the field of coal carbonization reveals the great importance, especially in small-scale tests, of applying the principles of specifications, tests, and facilities already established in matters concerning properties of materials. Throughout its history, the coal carbonization industry, in effect, has handled coal as an engineering material, and has appreciated the importance of stresses and strains developed during the coking period. Very soon after by-product coke ovens first came into use, operators observed that different ovens made different kinds of coke from the same coal. In 1895, Fulton (**1**)[2] considered that "the pressure of the charge of coal in the narrow oven compressed the cell structure of its coke making it more dense than does the broad oven with shallow charges." In addition, it was observed then and thereafter that some coals would develop coking pressure enough to break down the walls and roofs of coke ovens. In this connection, Denig (**2**) writes that "probably more ovens have been damaged by the use of dangerously expanding coals than for any other reason." Here, as in other fields of engineering, it can be seen that the nature of stresses and strains is complex, and that the pressure, volume, and structural changes developed during carbonization may vary over a wide range depending not only upon the kind of coal, but also on the type of equipment, and the carbonizing conditions. Moreover, where using small-scale engineering models it can be seen that it is necessary to reproduce in the model the coke that is produced in plant ovens, and also to reproduce the progressively changing properties from the beginning to the end of the coking period. It is also evident that both stress and strain are produced by a third condition; namely, the coking process. This kind of problem, however, is often met with in widely diversified fields of engineering; it involves principles that remain the same, no matter where they may be used since they are already established in connection with specifications, tests, and facilities concerning properties of materials.

In this field, there is one curious situation which should be recognized; namely, that engineers do not seem to have fully exploited the usefulness of the idea that coal, for the purposes of the appended procedure, may be classified as an engineering material. While the material being coked in a coke-oven seems far removed from steel and other materials used in a variety of structural conditions, experimental work (**3, 4, 5, 6, 7, 8, 9, 10**) has demonstrated the practical value of applying the same essential principles notwithstanding the widely diversified fields. For instance, the usefulness of applying the idea of compressive strength to material within the coke oven during the period of coking was discussed in 1938 as follows (**7**): Mills (**11**) writes about pieces of concrete sawn from a sidewalk after 20 yr. service which showed an expansion of 0.05 per cent when placed in water and the same shrinkage after subsequent drying in air. He explained "that if the concrete were restrained so that no volume changes could take place, the resultant stresses introduced, considering the modulus of elasticity to be 2,000,000 psi. would amount to about 1000 psi., a stress probably equal to at least half the ultimate strength if in compression." A portion of this concrete cut to fit snugly and inserted into a cold coke oven would be restrained so that any tendency to expand laterally would cause a side thrust against each wall and would introduce a corresponding stress in the concrete. Thus, if the originally dry concrete be soaked with water, one would expect the side thrust to increase, and if the walls could stand it, the thrust would continue increasing up to the breaking strength where failure of the concrete would occur. In this hypothetical case, the maximum side thrust would amount to about 1000 psi., and we know that it could not possibly exceed the ultimate strength of the concrete.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this discussion, see p. 320.

The stress-strain curve naturally may be used to indicate the characteristics of the side thrust, and it obviously is useful to have the values for modulus of elasticity, the strain due to wetting, and the ultimate strength in compression. Little imagination is required to substitute for the piece of concrete, the material being coked in a coke-oven; recognize that in the latter case, the tendency to expand laterally is the result of the coking process; that stresses are introduced whenever an expanding material is contained in a coke oven during the period of coking; that where the compressive strength is exceeded, the material will fracture and move upward in the oven; and, that so long as the charge is free to move upward, the maximum side thrust cannot possibly exceed the ultimate compressive strength of the charge. With such considerations as a basis, therefore, the attached method handles the material contained in the small-scale model as a structural engineering material subjected to stresses and strains resulting from the coking process. The procedure should be useful where the interested parties have agreed that for their purpose, it is necessary to investigate matters such as the effect of vertical expansion and height of charge on the lateral pressure developed during carbonization.

To handle the material being coked in a coke oven as an engineering material is not so necessary in the case of full-scale tests. These tests should automatically provide for factors such as depth of charge, bulk density, compressibility characteristics, and ultimate strength throughout the period of coking. On the other hand, where making small-scale tests, in order to get relatively accurate and reliable information, it is necessary to apply the laws of similitude which have their foundation in the rigorous logic of physical, chemical, mathematical and engineering fundamentals. Karpov (12) for instance, tells that "there is a certain relationship between the behavior of the model and that of its prototype which forms a part of a set of similarity conditions. The similarity conditions cover the whole range of the model-prototype relationship and may become very complicated. In designing and building a model, the main problem is to obtain the simplest set of similarity conditions consistent with the purpose and practicability of the model, so that from measurements made on the model the exact behavior of the prototype, under different service conditions may be determined." Although Karpov worked in a remote field—on models of Calderwood and Boulder dams—his work became the basis of experimental work (**3, 4, 5, 6, 7, 8, 9, 10**) which has demonstrated that representative samples of coal may be coked in small-scale models of the coke oven, satisfactorily reproducing the industrial-scale conditions, and handling the material in the model during the period of coking in the same way that testing materials laboratories handle other structural engineering materials. This experimental work is summarized as follows:

Early in 1932, the Massachusetts Gas Companies started investigating the efficacy of a variety of methods for the determination of the expansion of coals and coal blends, with the ultimate object in view of ascertaining the nature and magnitude of forces believed to be generated by expanding coals during high temperature carbonization in coke ovens. A paper (**3**) published in 1935 discussed the methods then available, discussed the mechanism of carbonization and expansion in coke ovens, presented an outline of tests made at Everett, Mass., and described a device for the determination of expansion and pressures developed during carbonization of coal. The purpose of this and subsequent work (**3, 4, 5, 6, 7, 8, 9, 10**) is expressed in Section 1 (*a*) of the appended "Proposed Method of Test for Pressures, Strains, and Other Properties Developed During Carbonization of Coal." It is hoped that making it available will be useful in the present review of facilities and methods for the determination of pressures and other properties developed during the carbonization of coal.

REFERENCES

(**1**) J. Fulton, "Cokes," International Textbook Co., Scranton, Pa. (1906).

(**2**) F. Denig, Annual Convention, Assn. Iron and Steel Eng. (1938).

(**3**) V. J. Altieri, "Coal Expansion," Production and Chemical Conference, Am. Gas Assn. (1935).

(**4**) V. J. Altieri, "Measurement of the Expansion of Coal During Carbonization," *Proceedings*, Am. Gas Assn., p. 812 (1935).

(**5**) R. E. Zimmerman, "The Determination of the Relation Between Test Cokes and Industrially Produced Cokes Prepared from the Same Coal Blends," Northeastern University Thesis, Chemical Engineering (1936).

(**6**) P. F. Noble, "An Improved Coal Expansion Tester," Northeastern University Thesis, Chemical Engineering (1937).

(**7**) V. J. Altieri, "Coal Expansion During Carbonization in Engineering Models of a Coke-Oven," Production and Chemical Conference, Am. Gas Assn. (1938).

(**8**) V. J. Altieri, "Stresses and Strains Generated During Carbonization," Production and Chemical Conference, Am. Gas Assn. (1940).

(**9**) J. K. Swiniarski, M. C. Cryan, and V. J. Altieri, "Selection of Coals for War-Time Carbonization by Means of an Improved Engineering Model of the Coke-Oven," Production and Chemical Conference, Am. Gas Assn. (1942).

(**10**) R. B. Cox, "Plastic Properties of Coal," Northeastern University Thesis (1936).

(**11**) A. P. Mills, "Materials of Construction," John Wiley and Sons, Inc., New York, N. Y., p. 9 (1931).

(**12**) A. V. Karpov, "Materials for Use in Structural Engineering Models, with Special Reference to Rubber Compounds," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 663 (1934).

(**13**) Report of Subcommittee on New Developments, *Proceedings*, Am. Gas Assn. (1940-1941).

(**14**) Report of Subcommittee on New Developments, *Proceedings*, Am. Gas Assn. (1941-1942).

PROPOSED METHOD OF TEST FOR PRESSURES, STRAINS, AND OTHER PROPERTIES DEVELOPED DURING CARBONIZATION OF COAL

1. *Scope.*—(*a*) This method of test is intended for use in making preliminary coking tests, the results of which can be used as a basis for the selection of coals, coal blends, and operating conditions, so that stresses and strains developed during carbonization will not cause abnormal operation retarding production, nor cause immediate or deferred damage under the conditions of use.

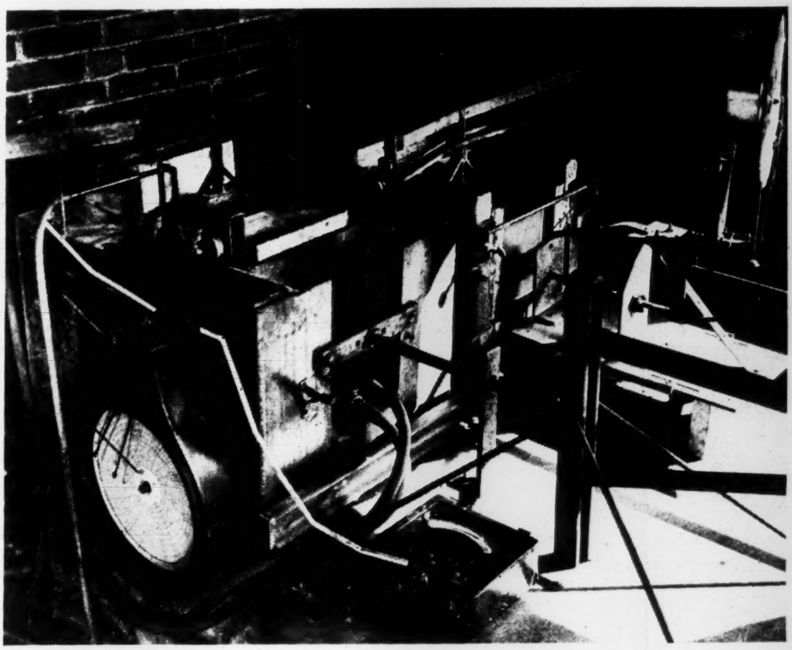
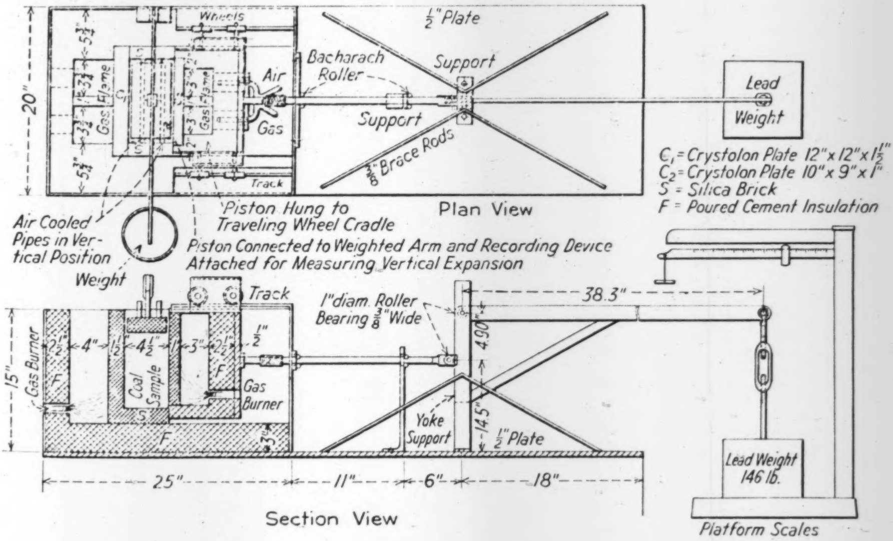


FIG. 1.—Coke Oven for Measuring Stresses and Strains Generated During Carbonization of Coal.



Wheels
$\frac{1}{2}$" Plate
20"
$5\frac{3}{4}$"
$3\frac{1}{4}$"
Gas Flame
Gas Flame
Air
Gas
Bacharach Roller
Support
Support
$\frac{3}{8}$" Brace Rods
Lead Weight
Track
Piston Hung to Traveling Wheel Cradle
Plan View
Air Cooled Pipes in Vertical Position
Piston Connected to Weighted Arm and Recording Device Attached for Measuring Vertical Expansion
Weight
Track
1" diam. Roller Bearing $\frac{3}{8}$" Wide
38.3"
4.90"
14.5"
15"
Gas Burner
$2\frac{1}{2}$"
4"
$1\frac{1}{2}$"
$4\frac{1}{2}$"
1"
3"
$2\frac{1}{2}$"
$\frac{1}{2}$"
F
Coal Sample
S
Gas Burner
3"
Yoke Support
$\frac{1}{2}$" Plate
Lead Weight 146 lb.
25"
11"
6"
18"
Section View
Platform Scales

C_1 = Crystolon Plate 12" x 12" x $1\frac{1}{2}$"
C_2 = Crystolon Plate 10" x 9" x 1"
S = Silica Brick
F = Poured Cement Insulation

FIG. 2.—Apparatus for Measuring Stresses and Strains Generated During Carbonization of Coal.

(*b*) The method is intended for use where factors such as amount of coal available, or, time, man-power, and expense, prevent the application of large, or full-scale oven tests. The method covers the production of coke by means of a small-scale model coke oven where it is required to simulate coke oven operations and evaluate the following:

(*1*) Pressures developed during the coking period,

(*2*) Strains developed during the coking period,

(*3*) Compressibility characteristics of the charge at different times during the coking period, and

(*4*) Evaluation of cokes produced from different raw materials under different conditions of test.

2. *Nature of Test.*—(*a*) This method measures the lateral pressure while permitting not more than 0.001 in. of wall movement, and measures vertical strains while controlling the vertical load so as to maintain a stress of 2 psi. on the uncoked material between the plastic zones.

(*b*) The material being coked undergoes volume changes. When the material tends to expand, but is restrained so that no change of volume can take place, a thrust is exerted against the restraining surfaces and a corresponding stress is introduced in the material. When the compressive strength is exceeded, the charge will fracture and move upward in the oven; and so long as the charge is free to move upward, the maximum side thrust cannot possibly exceed the ultimate compressive strength of the charge.

NOTE: *Precaution.*—The nature of stresses and strains developed during carbonization is complex. It should accordingly not be assumed that the test may be used without limitations. Extensive investigations have demonstrated that the magnitude of the pressure and volume changes developed during carbonization may vary over a wide range depending not only upon the kind of coal, but also on the type of equipment, and the carbonizing conditions. Consequently, to get practical data, it is necessary to operate the test oven while conforming with essential similarity requirements such as moisture content, fineness, state of compaction, bulk density, and rate of heating. Furthermore, it is necessary to run the test and interpret data on the basis of coals whose acceptability is demonstrated by years of use.

3. *Definitions.*—When applicable, definitions of terms such as *stress, strain, compressive strength*, etc., shall conform with the Standard Definitions of Terms Relating to Methods of Testing (A.S.T.M. Designation: E 6).[1] Also for the purposes of this method, the following additional definitions shall apply:

Vertical Stress.—The stress calculated on the basis of the cross-section in a horizontal plane of the uncoked material between the plastic zones.

NOTE.—During the period of coking there is a progressive decrease in the cross-section of the uncoked material between the plastic zones. The required vertical stress is maintained by means of water in a pail having an orifice permitting the escape of water at the prescribed rate.

Coking Pressure.—The lateral stress, or side thrust, developed before the time of coalescence of the two plastic zones.

Coalescence Pressure.—The maximum lateral stress, or side thrust, developed during the time of coalescence of the two plastic zones.

Lateral Strain.—The strain measured along the width of the oven and perpendicular to the hot walls.

Vertical Strain.—The strain measured along the depth of the charge and perpendicular to the floor of the oven.

4. *Apparatus.*—The apparatus shown in Fig. 1 shall be constructed in accordance with Fig. 2, and shall consist of the following:

(*a*) *Test Oven.*—The test oven shall be assembled as follows: The $\frac{1}{8}$-in. steel shell, 15 by 20 by 25 in. shall be welded on the 20 by 60 by $\frac{1}{2}$-in. steel plate and anchored to the concrete pad or base as shown in Fig. 1. Refractory material[2] shall be poured into the shell in which wooden forms have been placed to form the oven chamber, fixed heating flues, and burner ports. The silicon carbide plate C_1, (Fig. 2) shall be cemented in place. The end walls and floor of the oven shall be lined with silica brick forming an oven 9¼ by 10 by 4½ in. The width may be varied from 1 to 9 in.

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[1] 1942 Book of A.S.T.M. Standards, Part III, p. 849.

[2] Firecrete or equivalent refractory material is satisfactory.

NOTE.—The test oven (Fig. 2) is an engineering model of the coke oven. It is not directly applicable in tests relating to other apparatus as, for example, vertical retorts.

(*b*) *Movable Wall.*—A movable wall consisting of a ⅛-in. steel frame 7 by 11 by 11 in., the back of which shall be reinforced with a ½-in. steel plate. Refractory material[2] shall be poured into the frame in which wooden forms are placed to form the heating flues, burner ports, and anchorage for positioning of the silicon carbide plate 10 by 9 by 1 in. To minimize friction, the movable wall shall be suspended by means of a wheel cradle assembly welded to the sides of the frame. The outer surface of the two wheels on each side shall be grooved to fit a track (½-in. round steel rod) welded to a base plate resting on top of the oven walls. The wheels may be installed by using greased bearings or suitable roller bearings.

(*c*) *Piston.*—A piston 9 by 4 by 3 in. shall be cast with refractory material,[2] coated with sodium silicate and fired to produce a smooth and hard surface. Two ⅛-in. iron pipes shall be cast in the piston 1 in. apart, in the lower part, running the length of the piston as shown in Figs. 1 and 2 and shall be air-cooled to prevent top coking of the charge during carbonization. The piston shall be connected to a weighted lever and a recording device attached for measuring the vertical expansion or contraction during coking. A vertical loading of 2 psi. shall be maintained on the material between the two plastic zones, by means of water in a pail having an orifice permitting the escape of water at a prescribed rate.

(*d*) *Gas-Air Pressure Burners.*—Two burners made from standard ½-in. iron pipe shall be set at an angle against silica deflectors to prevent hot spots at the base of the flues.

(*e*) *Pressure Indicator.*—The movable wall shall be connected with a 1-in. diameter steel rod carrying a 1-in. roller bearing wheel connected with the lateral lever as shown in Fig. 1. The lateral lever, made from steel ½ by 1½ in., and a 1-in. roller bearing imbedded in the fulcrum, shall be connected by means of a turnbuckle to a lead weight (146 lb.) set on platform scales of 500-lb. capacity and graduated in ¼-lb. subdivisions. The yoke support, reinforced with four ⅜-in. brace rods, shall have holes drilled in the upper section to permit adjustment of the lever ratio so that scale reading may be expressed in whole numbers.

5. *Sample.*—A representative gross sample (Note 1) shall be collected and prepared in accordance with the Standard Method of Sampling Coal for Analysis (A.S.T.M. Designation: D 21).[3] The sample shall be crushed to a size similar to that used in plant production (usually 80 to 85 per cent passing a ⅛-in. sieve). A sample of 11¼ lb. shall be used for the test in the 4½-in. oven. The bulk density of the sample shall be 55 ± 1 lb. per cu. ft. after tamping and puddling (Note 2).

NOTE 1.—The sample in general shall conform with essential similarity requirements, making moisture content, fineness, state of compaction, and bulk density simulate the conditions encountered in industrial or commercial practice.

NOTE 2.—While many crushed coals under conditions of use show a bulk density of about 55 lb. per cu. ft., there are some which naturally slump under apparently the same conditions and show 65 lb. per cu. ft. or more. It is advisable, therefore, to estimate or determine the initial bulk density under the conditions of use, and to obtain bulk density by air drying or otherwise conditioning the sample, or, by tamping and puddling.

6. *Procedure.*—(*a*) The oven shall be assembled as shown in Figs. 1 and 2, but the turnbuckle shall be left disconnected. Insulating fire bricks[4] shall be placed on the floor of the oven and against the end walls. The heat shall be turned on so as to bring the temperature of the hot walls to 1700 F. within 1¾ to 2 hr. at which time the heat shall be so adjusted as to maintain this temperature. The turnbuckle shall be connected and adjusted until the system is

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[3] 1942 Book of A.S.T.M. Standards, Part III, p. 11.

[4] Nonpareil or equivalent insulating fire brick are satisfactory.

balanced at 148 lb., scale reading. The system then shall be rebalanced every 10 min. until thermal equilibrium is established.

(*b*) The recording strainometer shall be prepared for use. The main gas and air valves shall be shut off; the last setting of the burner valves left undisturbed; and the insulating fire bricks removed. The sample shall be poured into the oven and puddled and tamped to a depth giving the desired bulk density (Section 5, Note 2).

(*c*) The piston shall be placed on the charge, and the lever shall be connected with the recording strainometer; the pail weighted with water shall be suspended at the end of the lever, and the orifice opened to permit escape of water so as to maintain a vertical stress of 2 psi. The bulk density at this time shall be measured by observing the distance between the point at the end of the lever and a predetermined point indicating a bulk density of 55 lb. per cu. ft. The charging procedure up to this time shall require not more than 3 min.

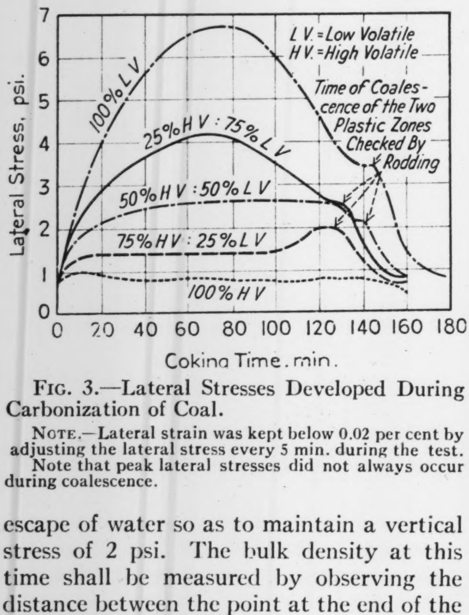

FIG. 3.—Lateral Stresses Developed During Carbonization of Coal.

NOTE.—Lateral strain was kept below 0.02 per cent by adjusting the lateral stress every 5 min. during the test. Note that peak lateral stresses did not always occur during coalescence.

(*d*) The main gas and air valves shall be opened, and heat shall be applied as described in Paragraph (*a*). The cooling flues of the piston shall be connected with the air supply and the flow or air shall be maintained so as to prevent excessive top coking and aid the unidirectional flow of heat. For this method, 3 cu. ft. per min. is satisfactory.

(*e*) Measurement of pressure developed and temperature observations at the cauliflower in the coke surface shall be made every 10 min. for the first 100 min., and every 5 min. thereafter until coking is completed. The test shall be stopped at the end of 2¾ hr. and the coke withdrawn, quenched, dried, and weighed. Where it is required to study the shape, fracture, and other features of the coke charge, the test shall be stopped at the end of 2¾ hr. and the charge shall be examined after cooling to room temperature. Samples shall be taken and the data recorded as described in Section 7.

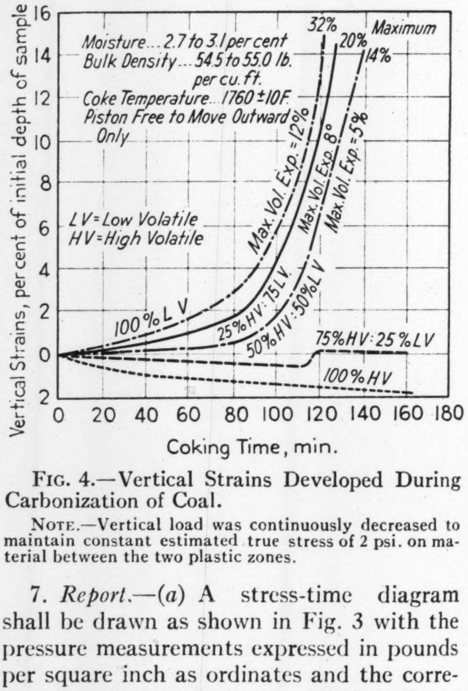

FIG. 4.—Vertical Strains Developed During Carbonization of Coal.

NOTE.—Vertical load was continuously decreased to maintain constant estimated true stress of 2 psi. on material between the two plastic zones.

7. *Report.*—(*a*) A stress-time diagram shall be drawn as shown in Fig. 3 with the pressure measurements expressed in pounds per square inch as ordinates and the corresponding times as abscissas. The line drawn through these points summarizes the behavior of the sample with respect to the coking pressures and the coalescence pressures developed by the coking process under the conditions of the test.

(*b*) Vertical strains (as taken from the strainometer chart Section 6 (*b*)), and temperatures of the hot walls adjacent to the coke cauliflower surface, shall be plotted as ordinates with the corresponding times as abscissas.

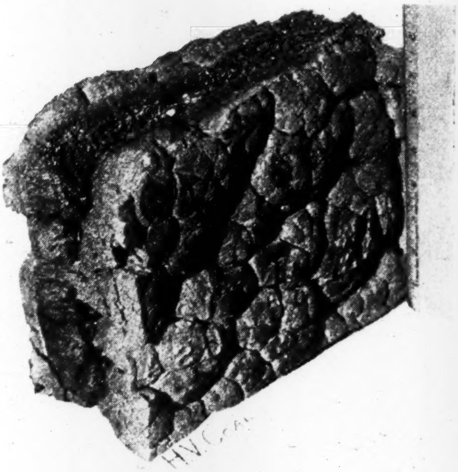

FIG. 5.—Appearance of High-Volatile Coal After Carbonization.

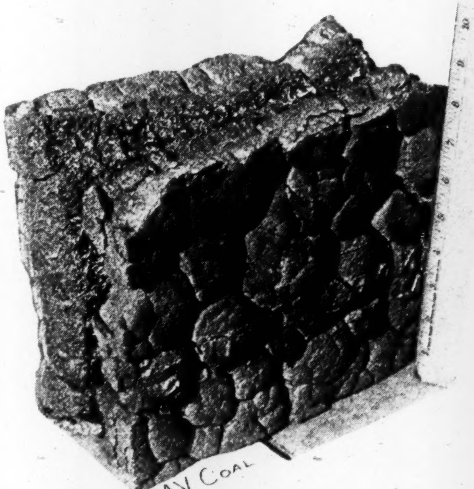

FIG. 6.—Appearance of Medium Volatile Coal After Carbonization.

(*c*) Additional data to suit the purpose of the work shall be recorded.[5] Such data shall be secured in accordance with all the requirements of good laboratory technique.

8. *Reproducibility of Results.*—The limit of accuracy of this method of test depends upon the attention which is given to details such as sampling and testing technique, and reproducibility of the test conditions. Consideration shall be given to factors such as moisture content, fineness, state of compaction, rate of heating, and escape of semi-fluid as well as gaseous material. With care and proper attention to details, duplicate determinations of the maximum lateral pressure should not differ by more than 10 per cent.

NOTE.—For a proper test the materials as well as the conditions met with in practice must be reproduced satisfactorily. It is essential, therefore, to select the test conditions so as to produce in the test oven substantially the same sort of coke as would be produced on a commercial or industrial scale. With care and proper attention to details, this method of test should produce coke which possesses a high degree of correlation with coke produced in large scale ovens. (See Figs. 5, 6, and 7.)

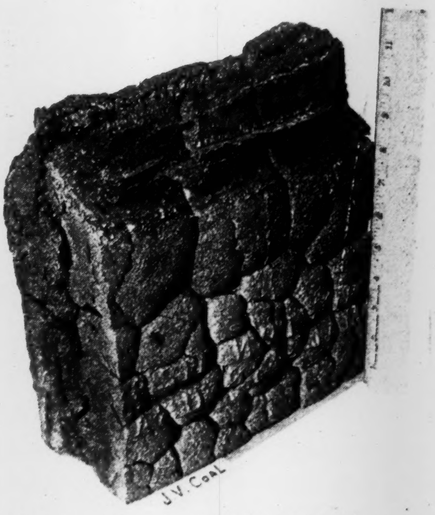

FIG. 7.—Appearance of Low-Volatile Coal After Carbonization.

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[5] A special report form for use in recording information obtained with this method of test has been prepared and is available from A.S.T.M. headquarters, 260 S. Broad St., Philadelphia 2, Pa.

REPORT OF COMMITTEE D-6

ON

PAPER AND PAPER PRODUCTS

Committee D-6 on Paper and Paper Products held one meeting during the year, on June 23, 1942, in Atlantic City, N. J.

Cooperative relations are being continued between Committee D-6, and other A.S.T.M. committees, as well as other standardizing bodies. Since the last meeting, R. D. Bonney has been appointed as the representative of Committee D-6 on A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials. For purposes of information, the preceding table is published, giving the exchange of representatives on these various committees and standardizing groups.

Committee	D-6 Representative on Committee	Representative on Committee D-6
A.S.T.M.		
C-16 on Thermal Insulating Materials	C. C. Heritage	....
D-8 on Bituminous Waterproofing and Roofing Materials	R. D. Bonney	M. R. Beasley
D-9 on Electrical Insulating Materials	J. M. Finch	R. W. Chadbourn
Joint D-6 and D-9 Committee on pH of Paper	Helen Kiely M. S. Kantrowitz J. M. Finch	B. L. Clark M. Guarnier C. F. Hanson
D-10 on Shipping Containers	....	Edward Dahill
Committee E-1 on Methods of Testing:		
Tech. Comm. III on Particle Size and Shape	W. R. Willets	....
Tech. Comm. X on Conditioning and Weathering	L. S. Reid	....
Tech. Comm. XIII on Hydrogen Ion Determinations	M. S. Kantrowitz	....
Joint A.S.T.M.-T.A.P.P.I. Committee on Paper Testing Methods	G. H. Harnden R. C. Griffin	P. F. Wehmer B. L. Wehmhoff
T.A.P.P.I. Tech. Assn. of Pulp and Paper Industry	....	R. G. MacDonald
N.A.P.A. National Assn. of Purchasing Agents	....	C. L. Sheldon

During the year, the following changes and additions in membership have been approved:

New Member:

Hubbel Lathrop, Western Electric Co.

New Representatives:

R. W. Sawyer, replacing C. P. Kirchen, Hollingsworth & Vose Co.

C. E. Hrubesky, replacing M. Heinig, U. S. Forest Products Lab.

R. C. Sproull, replacing R. Hemphill, General Electric Co.

Resignations:

Henry Chequer, Jr., Metropolitan Life Insurance Co.

Gaylord Container Corp., H. L. Bode

R. W. McKinley, Arthur D. Little, Inc.

Committee D-6 now has three subcommittees: Subcommittee I on Paper Testing Methods, consisting of 24 members; Subcommittee II on Significance of Test Methods, consisting of 20 members; and Subcommittee IV on Fiberboard and Fiberboard Containers, consisting of 23 members.

At the present time, Committee D-6 consists of 64 members, of whom 53 are voting members; 23 are classified as producers, 20 as consumers, 16 as general interest, and 5 as consulting members.

I. NEW TENTATIVE STANDARDS

The committee recommends that the following new methods, prepared by Subcommittee I, be accepted for publication as tentative, as appended hereto:[1]

Tentative Methods of Test for:

Degree of Staining of Paper by Alkali,
Turpentine Test for Grease Resistance of Paper,
Wire and Felt Sides of Paper,
Surface Wettability of Paper (Angle-of-Contact Method),
Kerosine Number for Roofing and Flooring Felt by the Vacuum Method,
Resistance of Paper to Passage of Air.

II. REVISION OF TENTATIVE STANDARD

Tentative Method of Qualitative Examination of Mineral Filler and Mineral Coating of Paper (**D 686 - 42 T**).[2]—The committee is proposing the following revisions in this tentative method and recommends that as revised it be continued as tentative:

Section 2.—After the present Paragraph (*o*), add a new Paragraph (*p*) to read as follows, relettering the subsequent paragraphs accordingly:

> (*p*) *Sodium Carbonate* (Na_2CO_3).—C.P., powdered.

Section 4 (*c*).—Change to read as follows by the addition of the italicized words and the omission of the word in brackets:

> (*c*) Ignite a test specimen in accordance with the procedure described in the Standard Method of Test for Ash Content of Paper and Paper Products (A.S.T.M. Designation: D 586) of the American Society for Testing Materials. Divide the ash into *three* [two] portions, each of which shall contain not less than 0.05 g. of mineral matter, and place *two portions* in *separate* 250-ml. beakers.

Section 4 (*d*).—Change item (*1*) from its present form: namely,

> (*1*) Considerable undissolved matter indicates the presence of barium sulfate, calcium sulfate (which may be originally present as such or formed by the action of H_2SO_4 on the sulfite of carbonate), aluminum silicates, magnesium silicates, or more than one of these substances. If the solution is clear, aluminum and magnesium silicates are absent, and possibly barium sulfate. (Barium sulfate is partially soluble in H_2SO_4 (sp. gr. 1.84) and may be present in small quantity even if the solution is clear.)

to read as follows:

> (*1*) Considerable undissolved matter indicates the presence of aluminum and/or magnesium silicates, and/or diatomaceous earth. If the solution is clear, aluminum or magnesium silicates and diatomaceous earth are absent. (Calcium and barium sulfates will not be in an undissolved state unless the sample being tested is more than 0.5 g.)

Change item (*4*) to read as follows by the addition of the italicized words:

> (*4*) If there is a residue *when the test sample is more than 0.5 g.*, or precipitate from the treatment of the ash with H_2SO_4 (Paragraph (*d*)), filter, wash with H_2SO_4 (5:95), and make a flame test with the platinum wire; a green flame indicates the presence of barium, a red flame indicates calcium, and a yellow to colorless flame indicates aluminum or magnesium silicates, or both.

Section 4 (*g*).—Change from its present form: namely,

> (*g*) Remove the residue from the initial HCl treatment of the ash (Paragraph (*e*)) to a charcoal block and moisten with the $Co(NO_3)_2$ solution. Aluminum will give a fugitive blue coloration upon heating with a blowtorch flame.

to read as follows:

> (*g*) Mix the third portion with an excess of sodium carbonate on a charcoal block, and moisten with a small amount of cobalt nitrate solution. Aluminum will give a permanent blue coloration upon heating with a blowtorch flame. (Care should be taken not to mask the blue coloration by the use of an excess of cobalt nitrate solution.)

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[1] These methods were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, pp. 210, 212, 227, 223, 214, and 220, respectively.

[2] 1942 Book of A.S.T.M. Standards, Part III.

Section 5 (f).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*f*) The residue from the portion of the ash treated with H_2SO_4 (sp. gr. 1.84) may be clay, talc, *diatomaceous earth* [barium sulfate], or a mixture of these substances. A positive test for aluminum indicates that clay was used.

III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following four tentative standards[2] be approved without change for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of:

Conditioning Paperboard, Fiberboard, and Paperboard Containers for Testing (D 641 – 41 T),
Compression Testing of Corrugated and Solid Fiber Boxes (D 642 – 41 T),
Test for Folding Endurance of Paper (D 643 – 41 T), and
Test for Thickness of Paper and Paper Products (D 645 – 41 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 53 voting members; 43 members returned their ballots with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Paper Testing Methods (W. R. Willets, chairman) has held two meetings during the year and has continued its critical study of various methods of test that have been approved by the Technical Association of the Pulp and Paper Industry and is also active in developing new methods of test. The subcommittee prepared seven new methods of test that are being recommended for publication as tentative, as mentioned earlier in this report.

The four tentative methods being recommended for adoption as standard, are also under the jurisdiction of this subcommittee.

In addition, the subcommittee recommends that the Tentative Method of Test for Opacity of Paper and Paper Products (D 589 – 40 T) be retained as tentative. This method at present determines opacity by the contrast ratio. It is not suitable, however, for measuring printing opacity and, therefore, its adoption as standard is not recommended at this time. Considerable work is now being carried on in an effort to revise this method as soon as possible to include a test procedure for printing opacity.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Test for Degree of Staining of Paper by Alkali	26	0	17
Turpentine Test for Grease Resistance of Paper	29	0	14
Test for Wire and Felt Sides of Paper	30	0	13
Test for Surface Wettability of Paper (Angle - of - Contact Method)	24	0	19
Test for Kerosine Number for Roofing and Flooring Felt by the Vacuum Method	23	0	20
Test for Resistance of Paper to Passage of Air	30	0	13
II. REVISION OF TENTATIVE STANDARD			
Method of Qualitative Examination of Mineral Filler and Mineral Coating of Paper (D 686 – 42 T)	26	0	17
III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Method of Conditioning Paperboard, Fiberboard and Paperboard Containers for Testing (D 641 – 41 T)	31	0	12
Method of Compression Testing of Corrugated and Solid Fiber Boxes (D 642 – 41 T)	24	0	17
Test for Folding Endurance of Paper (D 643 – 41 T)	32	0	11
Test for Thickness of Paper and Paper Products (D 645 – 41 T)	33	0	10

Methods on the following subjects are being given consideration by Subcommittee I:

Fiber Composition of Paper,
Ply Adhesion of Paper,
Wax Pick Test for Surface Strength of Paper,

Stretch of Paper and Paperboard,
Tensile Breaking Strength of Paper and Paper Products,
Wet Tensile Strength of Paper,
Erasing Properties of Paper,
Edge Tear of Paper,
Curl of Paper,
Stiffness of Paper,
Hydrogen Ion Concentration (Unbuffered and Neutral Papers),
Gloss, and
Water Absorption of Paper.

In addition to the above, work has been completed on the following proposed methods, which have been referred to the Joint A.S.T.M.-T.A.P.P.I. Committee for action:

Time of Penetration by Water of Sized Paper and Paper Products,
Measuring Bursting Strength of Paper,
Hydrogen Ion Concentration of Paper Extracts,
Determining the Effect of Heating on Folding Endurance of Paper,
Castor Oil Method for Measuring Printing Oil Penetration of Paper, and
Water Vapor Permeability of Paper.

In addition to the above methods, Subcommittee I referred back to the Joint A.S.T.M.-T.A.P.P.I. Committee a method for dirt in paper, as being of no interest to the committee.

Subcommittee II on Significance of Test Methods (C. C. Heritage, chairman) held one meeting during the year at which a plan was laid out to expedite the completion of the monograph covering the significance of tests of paper, and definitions of terms, nomenclature, and properties of paper. The monograph is practically completed and parts of the manuscript are now being given a final reading by members of the committee. It is expected that it will be ready for publication this fall.

Subcommittee IV on Fiberboard and Fiberboard Containers (R. L. Beach, chairman) has held no meetings during the year as the work of this subcommittee has had to be temporarily suspended during this period because the individual members directly concerned with the investigations in progress are devoting their entire time to the war effort.

The Advisory Committee has requested A.S.T.M. Headquarters to arrange for publication after the annual meeting of all the methods of test under the jurisdiction of Committee D-6. This special compilation will include the 22 methods now published as standards or tentative standards, if approved, and the 7 new methods being recommended in this report for publication as tentative.

This report has been submitted to letter ballot of the committee, which consists of 53 voting members, 43 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. S. REID,
Chairman.

G. H. HARNDEN,
Secretary.

REPORT OF COMMITTEE D-8

ON

BITUMINOUS WATERPROOFING AND ROOFING MATERIALS

Committee D-8 on Bituminous Waterproofing and Roofing Materials held one meeting during the year, in Atlantic City, N. J., on June 23, 1942.

Subsequent to the 1942 Annual Meeting, Committee D-8 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:

Asphalt Siding Surfaced with Coarse Mineral Granules (D 699 - 42 T),

Revision of Tentative Specifications for:

Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 - 41 T), and

Revision of Tentative Methods of:

Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (D 228 - 37 T).

These recommendations were accepted[1] by Committee E-10 on November 9, 1942, and the new and revised tentative standards appear in the 1942 Book of A.S.T.M. Standards, Part II.

Subcommittees of Committee D-8 have under review proposed revisions of a number of existing specifications which will probably be presented to the Society through Committee E-10 during the coming year.

EDITORIAL CHANGE IN STANDARD

The committee recommends that the following editorial change be included in the Tentative Specifications for Asphalt for Dampproofing and Waterproofing (D 449 - 42 T):

> *Section 4.*—Change "Type A" to read "Type B" and change "Type B" to read "Type A," making a corresponding change in Table I on physical properties of asphalt, in order to conform with the type designations in the Standard Specifications for Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (D 450 - 41).

Committee D-8 requests that the remaining standards and tentative standards for which it is responsible be continued in their present form without revision.

This report has been submitted to letter ballot of the committee which consists of 61 voting members; 46 members returned their ballots, of whom 44 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. M. WEISS,
Chairman.

PRÉVOST HUBBARD,
Secretary.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-8 reported the following results of the letter ballot vote of a total of 46 ballots returned from a committee membership of 62: D 699, affirmative 39, negative 0, ballots marked "not voting" 7; D 249, affirmative 40, negative 0, ballots marked "not voting" 6; D 228, affirmative 40, negative 0, ballots marked "not voting" 6.

Editorial Note

Subsequent to the Annual Meeting, Committee D-8 presented to the Society through Committee E-10 on Standards the following recommendations under the emergency procedure:

Emergency Alternate Provisions in:

Standard Methods of Testing Bituminous Mastics, Grouts, and Like Mixtures (EA - D 147),

Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (EA - D 228),

Additional Emergency Alternate Provisions in:

Tentative Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica (EA - D 224a), and

Tentative Specifications for Asphalt Roofing Surfaced with Coarse Mineral Granules (EA - D 249a).

These recommendations were accepted by Committee E-10 on August 30, 1943, and the emergency provisions were published in ASTM Bulletin, No. 125, December, 1943, p. 65, and have been issued in the form of pink stickers for attachment to the specifications and methods to which they apply.

REPORT OF COMMITTEE D-9

ON

ELECTRICAL INSULATING MATERIALS

Committee D-9 on Electrical Insulating Materials held two meetings during the year and will hold a third during the Annual Meeting of the Society in June. Both of these meetings were held in New York, N. Y., on October 6 and 7, 1942, and on March 4 and 5, 1943. These meetings were held in conjunction with the meetings of Committee D-20 on Plastics because of the close cooperation between the two committees and the large number of members who serve on both committees.

At these meetings recommendations were made which resulted in new standards, additions to existing standards, and changes in some of the standards for which Committee D-9 is responsible. Projects which are still under way are mentioned later in this report.

Recommendations Accepted by Committee E-10 on Standards

Subsequent to the 1942 Annual Meeting, Committee D-9 presented to the Society through Committee E-10 on Standards, the following recommendations:

Revisions of Tentative Methods of:

Testing Molded Materials Used for Electrical Insulation (D 48 - 41 T),

Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 - 41 T), and

Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 - 40 T).

These recommendations were accepted[1] by Committee E-10 on August 24, 1942, and the revised tentative methods appear in the 1942 Book of A.S.T.M. Standards, Part III.

Committee D-9 and Committee D-20 on Plastics jointly submitted the following specifications to Committee E-10 during the year:

New Tentative Specifications for:

Phenolic Molding Compounds (D 700 - 43 T), and

Laminated Thermosetting Materials (D 709 - 43 T).

These specifications were accepted[1] by the Standards Committee on March 24, 1943, and April 5, 1943, respectively, and have been assigned the A.S.T.M. serial designations indicated above.[2]

Committee D-9 recently submitted to Committee E-10 proposed Tentative Specifications for Vulcanized Fibre which were accepted[1] on April 27, 1943, and issued under the A.S.T.M. designation D 710 - 43 T.[2]

Recommendations Affecting Standards

As the result of the year's work, the committee is submitting two new tentative standards, revisions of three tentative standards and tentative revisions of four standards, revision of a standard for immediate adoption, and is recommending the adoption of two tentative standards as standard, the adoption as standard of one tentative revision of standard, and editorial changes in two tentative standards. The standards affected are listed in Table I. The recommendations being presented are included in Appendix I, each proposed revision being accompanied by a brief reason for the revision.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-9 reported the following results of the letter ballot vote of a total of 68 ballots returned from a committee membership of 96: D 48, affirmative 35, negative 0, ballots marked "not voting" 33; D 150, affirmative 42, negative 1, ballots marked "not voting" 25; D 176, affirmative 26, negative 1, ballots marked "not voting" 41; D 700, 71 ballots returned: affirmative 26, negative 2, ballots marked "not voting" 43; D 709, 59 ballots returned: affirmative 27, negative 0, ballots marked "not voting" 32; D 710, 74 ballots returned: affirmative 24, negative 0, ballots marked "not voting" 50.

[2] These specifications have been published in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, pp. 280, 313, and 255, respectively.

These recommendations have been submitted to letter ballot of the committee which consists of 97 voting members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Tentative Standards			
Spec. for Orange Shellac, Pure Garnet and Button Lac	20	0	45
Spec. for Low- and Medium-Voltage Pin-Type Lime-Glass Insulators	20	2	43
II. Revisions of Tentative Standards			
Testing Molded Materials Used for Electrical Insulation (D 48 – 42 T)	35	0	30
Test for Saponification Number of Petroleum Products by Color-Indicator Titration (D 94 – 42 T)	24	0	41
Testing Varnished Cloths and Varnished Cloth Tapes Used In Electrical Insulation (D 295 – 38 T)	27	1	32
III. Revision of Standard, Immediate Adoption			
Testing Electrical Insulating Oils (D 117 – 42)	37	0	28
IV. Tentative Revisions of Standards			
Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 42)	38	0	27
Testing Laminated Tubes Used in Electrical Insulation (D 348 – 42)	34	0	31
Testing Laminated Round Rods Used in Electrical Insulation (D 349 – 42)	34	0	31
Testing Electrical Insulating Oils (D 117 – 42), omission of Section 26	31	0	30
V. Adoption of Tentative Standards as Standard			
Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 – 40 T)	25	0	43
Measuring Mica Stampings Used in Electronic Devices and Incandescent Lamps (D 652 – 41 T)	20	0	45
VI. Adoption as Standard of Tentative Revision of Standard			
Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 42)	38	0	27
VII. Editorial Changes in Tentative Standards			
Spec. for Phenolic Laminated Sheets for Radio Applications (D 467 – 40 T)	32	0	33
Spec. for Round Phenolic Laminated Tubing for Radio Applications (D 616 – 41 T)	33	0	32

Activities of Subcommittees

Since the last Annual Meeting of the Society, this committee has reorganized some of its work so that there are now thirteen subcommittees carrying on active work. The former Subcommittee VIII on Insulating Papers and Fabrics has been broken up into two subcommittees: Subcommittee VII on Insulating Fabrics under the chairmanship of R. W. Chadbourn, formerly chairman of the old subcommittee, and Subcommittee VIII on Insulating Papers under the chairmanship of C. T. Hatcher. The former Subcommittee VII on Electrical Tests is now designated Subcommittee XII. A new correlating Subcommittee XIII on Mechanical Tests has been formed under the chairmanship of F. S. Mapes.

Some of the projects now in progress are briefly mentioned in the following summary of subcommittee activities:

Subcommittee I on Insulating Varnishes, Paints and Lacquers (C. F. Hanson, chairman).—This subcommittee has recommended to Committee D-9 that the Specifications for Orange Shellac, Pure Garnet and Pure Button Lac,[3] prepared in cooperation with Committee

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[3] These specifications were accepted as tentative by the Society and have been incorporated in the revised Tentative Specifications for Orange Shellac and Other Lacs (D 237 - 43 T), see Editorial Note, p. 258. The revised specifications are published in the 1943 Supplement to Book of A.S.T.M. Standards, Part II, p. 135; Part III, p. 229.

D-1 on Paint, Varnish, Lacquer, and Related Products, be submitted to the Society for publication as tentative.

Work is still in progress to develop methods of test for deep drying varnishes and for acid and alkali resistance of varnishes. This study has been carried on for a number of years and there is still considerable work to be done before satisfactory test methods are evolved. A new section has been formed to prepare statements on significance of tests of the various test methods listed in the Standard Methods of Testing Varnishes Used for Electrical Insulation (D 115 - 41).

Subcommittee II on Molded Insulating Materials (W. A. Evans, chairman).—Work has been completed on the Tentative Specifications for Phenolic Molding Compounds which have been accepted by the Society and assigned the designation D 700 - 43 T. These specifications were a cooperative project under the joint sponsorship of Subcommittee II of Committee D-9 and Subcommittee VI of Committee D-20 on Plastics. They are being considered in connection with the work of the War Committee on Radio which was formed under the war standards procedure of the American Standards Association, at the request of the War Production Board, to set up war standards for components for military radio to help speed production of radio equipment for the Army and Navy, make replacement of parts easier, and conserve materials. This and other D-9 subcommittees are actively cooperating in this work.

A study is being made of the effect of location of the notch in the impact bar on the results of the impact test. The present test method calls for the location of the notch in the side of the specimen while much commercial data have been obtained with the notch in the face of the specimen. This matter is being investigated since it is of considerable importance in setting up detailed requirements in specifications.

Subcommittee III on Plates, Tubes, and Rods (G. H. Mains, chairman).—The subcommittee is actively cooperating with Committee D-20 on Plastics and with the War Committee on Radio in the preparation of specifications for plastics and electrical insulating materials in the form of sheets, rods, and tubes. Tentative Specifications for Vulcanized Fibre, prepared by this subcommittee, have been accepted and issued under the designation D 710 - 43 T. Tentative Specifications for Laminated Thermosetting Materials, prepared jointly with Committee D-20, have also been accepted and assigned the designation D 709 - 43 T. These specifications will be rapidly revised and improved where necessary to meet the needs of the Armed Forces. Review is being made of the values for electrical properties in the specifications for vinyl chloride acetate, vinylidene chloride, and other plastic sheet being prepared by Committee D-20.[4]

The coordinating work on mechanical test methods undertaken with representatives of Subcommittee I of Committee D-20 has resulted in revision in the tensile strength methods of the two committees to secure practical uniformity. Compressive and flexural strength test methods are also being studied to obtain greatest possible coordination.

The study of arc-resistance test methods to classify materials which react differently under low- and high-density arcs is being continued.

In cooperation with Subcommittee X, work is being conducted to revise the present conditioning procedure for specimens for mechanical tests to apply to thin materials ($\frac{1}{32}$ in. and under). Work is also being continued on methods for conditioning sheet materials for the various electrical tests.

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[4] See p. 402.

Correlation between the D-9 and D-20 methods on flammability of sheet materials is being studied; also the applicability of the D-20 method for thermal coefficient of linear expansion to various electrical insulating materials.

The subcommittee regrets the resignation of Dean Harvey from the chairmanship due to pressure of other work and expresses its appreciation for his splendid service for a period of over 20 years.

Subcommittee IV on Liquid Insulation (E. A. Snyder, chairman).—The activities of this subcommittee have been as follows:

(*1*) *Neutralization Number.*—Section A on Neutralization Number has finally completed several years of investigation of modified testing methods and electrometric titration methods. During this study they worked very closely with Committee D-2 on Petroleum Products and Lubricants on this same problem and a final recommendation is being made by Committee D-2 to adopt as standard the Tentative Method of Test for Neutralization Number of Petroleum Products by Color-Indicator Titration (D 663 – 42 T), and as an alternate method for those who have the equipment and prefer the Electrometric Titration Test, Tentative Method D 664 – 42 T. Supporting data for these recommendations have been submitted to the Society. The results of the final round-robin tests covering the use of the former Method of Test for Neutralization of Insulating Oils (D 188 – 41 T), and the new Titration Method (D 663 – 42 T), as well as the Electrometric Titration Method (D 664 – 42 T) are shown in Table II.

(*2*) *Sludge Test.*—The Tentative Methods of Test for Sludge Formation in Mineral Transformer Oil (D 670 – 42 T) have been in force for one year and Section B of Subcommittee IV is actively at work preparing test data on the correlation of sludge test values as compared with actual service of such oils. It is necessary to secure such information before it is possible to recommend specification limits on sludging. This work will be continued during the coming year.

(*3*) *Insulating Oils and Askarels.*—A revision is recommended in the scope and title of the Standard Methods of Testing Electrical Insulating Oils (D 117 – 42), as given in Appendix I. During the year the subcommittee has increased its scope to develop test methods for synthetic insulating liquids and there is published as information with this report Proposed Methods of Testing Askarels.[5] The subcommittee will continue this work during the coming year looking

TABLE II.—RESULTS OF NEUTRALIZATION NUMBER TESTS.

Laboratory	Method D188-41T	Method D663-42T	Method D664-42T
SAMPLE D9-1-42 (NEW OIL)			
1	0.03	0.03	0.00
2	0.02	0.03	0.00
3	0.03	0.03	0.03
4	0.02	0.02	...
5	0.03	0.02	0.01
6	0.01	0.01	...
7	0.01	0.01	...
Average	0.02	0.02	0.01
Average Deviation	0.007	0.007	0.01
SAMPLE D9-2-42 (USED OIL)			
1	0.14	0.13	0.06
2	0.13	0.13	0.13
3	0.14	0.12	0.07
4	0.13	0.12	...
5	0.13	0.12	0.09
6	0.10	0.11	...
7	0.10	0.10	...
Average	0.12	0.12	0.09
Average Deviation	0.016	0.007	0.02
SAMPLE D9-3-42 (USED OIL)			
1	0.48	0.42	0.46
2	0.45	0.45	0.46
3	0.45	0.50	0.34
4	0.52	0.36	...
5	0.48	0.42	0.37
6	0.43	0.46	...
7	0.39	0.34	...
Average	0.46	0.42	0.41
Average Deviation	0.03	0.04	0.05

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[5] See p. 353.

toward submittal of this method for publication as tentative.

(*4*) *Saponification Number.*—The subcommittee has approved the Tentative Method of Test for Saponification Number of Petroleum Products by Color-Indicator Titration (D 94 – 42 T), under the jurisdiction of Committee D-2, as the standard method for determining saponification number of insulating oils with the exception that the addition of 50 mm. of petroleum naphtha to the oil sample is eliminated from the procedure when testing insulating oil. Committee D-2 has approved this recommendation and is adding this statement to the method.

(*5*) *Resistivity and Power Factor Test.*—The Section on Resistivity and Power Factor has been revived and work has been started in cooperation with Section B of Subcommittee XII on Power Factor of Liquids under the chairmanship of K. C. Coutlee. It is hoped that the efforts of this cooperative work will develop a satisfactory test procedure for determining the power factor and resistivity of liquid insulating oils.

(*6*) *Gas Content of Oil.*—The section studying this problem has been working for several years and has now developed a Proposed Routine Method of Test for Gas Content of Insulating Oil which appears in Appendix II of this report as information. The section is continuing its activities to prepare a standard referee test. It is hoped that publication of the routine test will result in producing data and comments from users which will assist the section in its further work.

(*7*) *Dielectric Strength Test.*—This section has been continuing its activities during the year and as a result of its work a number of changes will be made in the tentative revision[6] of the Standard Methods of Testing Insulating Oils (D 117 – 42), which revision covers a new procedure for determining dielectric strength. This subcommittee recommends that the description of the procedure for preparation of sample which appears as Section 26 of the tentative revision be withdrawn.

(*8*) *Significance of Test.*—This section has now completed its work in defining the significance of tests for color, neutralization number, dielectric strength, mineral acids, pour point, flash point, specific gravity, free and corrosive sulfur, and viscosity when applied as indicated in the Standard Methods of Testing Electrical Insulating Oils (D 117 – 42). These new statements on significance of tests appear in Appendix III. The section is still working on the preparation of statements on the significance of tests for steam emulsion, power factor, resistivity, sludge, and free chloride.

(*9*) *Water in Insulating Oil.*—This section has been actively at work developing analytical test methods for determination of minute traces of water in insulating oil, and while satisfactory progress has been made to date, the section is not in a position to recommend any method for publication as information. It is hoped that such a method will be ready by the time of the next Annual Meeting.

Subcommittee V on Ceramic Products (L. J. Cavanaugh, chairman).—Members of this subcommittee have cooperated with the War Committee on Radio, under the sponsorship of the American Standards Association, in the preparation of American War Standards for Ceramic Insulating Materials.

The preparation of emergency specifications for communication and signal glass insulators is under way.

On the recommendation of the subcommittee, Specifications for Low- and Medium-Voltage Pin-Type Lime-Glass Insulators,[7] which in substance are identical with the Edison Electric Institute Specifications TD-51-1942, are being submitted for publication as tentative.

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[6] 1942 Book of A.S.T.M. Standards, Part III, p. 1592.

[7] These specifications were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 262.

A new section has been formed to prepare methods of test for high dielectric constant ceramic materials. This section will study the factors involved in measuring electrical properties of these materials.

Subcommittee VI on Solid Filling and Treating Compounds (R. H. Titley, chairman).—Work on solubility of solid filling and treating compounds in cable oil is still under consideration since no satisfactory accelerated test has been found. Additional tests are being conducted to find another means of evaluating this property and some progress is being made.

The Section on Waxes has presented to the subcommittee several test methods which are now being studied by the subcommittee and these should soon be available together with tentative specifications for these types of materials.

Subcommittee VII on Insulating Fabrics (R. W. Chadbourn, chairman).—This subcommittee is now carrying on the work formerly carried on by Division II of Subcommittee VIII. The same membership of that Division is continuing as this new Subcommittee VII on Insulating Fabrics.

Revisions are recommended in the Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 - 38 T) to make the test methods applicable to glass fabrics, as given in Appendix I. Additional tests will be recommended for glass fabrics as soon as they can be properly studied.

Existing elongation requirements for varnished cambric tapes are generally considered inadequate. This subject is being actively reviewed, and some modification of the present specifications may develop. The subcommittee is also investigating an alternative power factor test which eliminates the vacuum treatment. Several significance of tests statements are now in preparation.

Subcommittee VIII on Insulating Papers (C. T. Hatcher, chairman).—This is a new subcommittee made up largely of the membership of Division I of the former Subcommittee VIII. Work being carried on in conjunction with Committee D-6 on Paper and Paper Products is resulting in a method for determining the pH values of insulating papers. Correlation of the Tentative Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 41 T) with the methods under the jurisdiction of Committee D-6 is continuing with good prospects of complete agreement in methods very soon. It is recommended that Methods D 202 be continued as tentative because of this work which is being carried on at the present time. Some significance of test statements have been prepared and are now being considered by the subcommittee.

Subcommittee IX on Mica Products (E. O. Hausmann, chairman).—The project of preparing colored transparencies to be used for quality classification of natural micas is being developed. The chairman was instructed to negotiate for the first set of photographs which are to be checked by the subcommittee before continuing with the project. A revision of the methods for testing natural mica (D 351) is under consideration.

Existing specifications for natural and bonded mica are being reviewed in consideration of preparing A.S.T.M. specifications for these materials.[8]

The NEMA Standard for Manufactured Electrical Mica has been reviewed looking toward its submittal to the

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[8] See Editorial Note, p. 339.

American Standards Association for approval as American Standard. On the recommendation of the subcommittee, Sectional Committee C-59 on Electrical Insulating Materials took action to defer submittal of this standard to the ASA for the present time until the supply of mica meeting the specifications is more plentiful.

Subcommittee X on Conditioning (Robert Burns, chairman).—This subcommittee has two projects under active consideration at this time, the first of which is standardization of testing conditions for extremes of high and low temperatures as applied to plastics and insulating materials. The Government war service groups are requesting data on properties of materials when tests are made at extremes of temperature and humidity. This subcommittee is attempting to reduce the number of conditions at which these measurements are to be made to an irreducible minimum necessary to meet the requirements of the service groups. A second project being carried on is the development of equipment for providing air at high or low temperatures to chambers surrounding test equipment. This project is connected with the first project in that much of this extreme temperature testing must be done under conditions which are difficult to obtain in the average laboratory. This subcommittee is working with manufacturers of conditioning equipment in a joint attempt to provide the proper testing atmosphere for this work.

Subcommittee XI on Significance of Tests (H. L. Curtis, chairman).—This subcommittee is reviewing significance of test statements originating in various subcommittees. Nine such statements concerning properties of insulating oils have been prepared and submitted to Committee D-9 for approval. Some of the older statements appearing in the compilation of A.S.T.M. Standards on Electrical Insulating Materials are being reviewed in order to bring them up to date. Consideration is now being given to having these statements published in a form which would make them easily available to those interested in securing copies

Subcommittee XII on Electrical Tests (Gordon Thompson, chairman).—The Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 – 42 T) for which this subcommittee is responsible continues to receive thoroughgoing examination and criticism with the result that changes intended more carefully to define conditions or more precise measurements, are offered each year; the past year has been no exception to the prevailing trend, further revisions having been accepted by Committee E-10, as mentioned earlier in this report. Covering 31 pages of text, with mathematical appendix and bibliography this method constitutes practically a treatise on the subject. The subcommittee has been largely reorganized during the year, new section chairmen having been appointed in many cases and the section membership extensively readjusted. Not much round-robin testing by the sections has been done during the year but the sections have an extensive program of work laid out and as soon as time and men are available, considerable progress may be expected in measurements of insulation resistance and absorption phenomena, in measurements of insulating liquids, and in tests for both solid and liquid insulations at ultra-high frequencies.

Subcommittee XIII on Mechanical Tests (F. S. Mapes, chairman).—This subcommittee was organized during the year and the work of the group outlined. This will be a coordinating group whose particular task will be to coordinate the various mechanical test methods developed in the other D-9 subcommittees. The first meeting of this subcommittee was held in connection with the March meetings of Committee D-9 at which time several sections were set up to take care of the various activities.

This report has been submitted to letter ballot of the committee which consists of 97 voting members; 52 members returned their ballots, of whom 48 have voted affirmatively and 0 negatively

Respectfully submitted on behalf of the committee,

Myron P. Davis,
Chairman.
W. A. Evans,
Vice-Chairman.
W. A. Zinzow,
Secretary.

Editorial Note

Subsequent to the Annual Meeting, Committee D-9 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Methods of:

Measuring Dimensions of Rigid Rods Used in Electrical Insulation (D 741 – 43 T), and

Revision of Tentative Revision of:

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 43).

These recommendations were accepted by Committee E-10 on August 30, 1943, and the new tentative standard and revised tentative revision appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, pp. 260 and 527.

On September 24, 1943, Committee E-10 accepted proposed revisions in the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 – 41 T) recommended by Committee D-9 jointly with Committee D-20 on Plastics. The revised tentative methods appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 249, bearing the designation D 256 – 43 T.

Proposed Specifications for Natural Block Mica and Mica Films Suitable for Use in Fixed Mica-Dielectric Capacitors were accepted by Committee E-10 on November 20, 1943, for publication as tentative. The new tentative specifications have been assigned the designation D 748 – 43 T and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 273.

APPENDIX I

RECOMMENDATIONS AFFECTING STANDARDS FOR ELECTRICAL INSULATING MATERIALS

In this Appendix are given recommendations affecting certain standards and tentative standards covering electrical insulating materials which are referred to earlier in this report.[1] The standards and tentative standards appear in their present form in the 1942 Book of A.S.T.M. Standards, Part III.

New Tentative Standards

Tentative Specifications for Orange Shellac (Pure Garnet and Button Lac):[2]

These specifications outline the requirements for two types of lacs not covered by the Standard Specifications for Orange Shellac (D 237 – 41). It is recommended that these specifications be accepted for publication as tentative.

Tentative Specifications for Low- and Medium-Voltage Pin-Type Lime-Glass Insulators:[3]

These specifications, prepared by the Standards and Specifications Group of Edison Electric Institute in cooperation with the members of a section of Subcommittee V of Committee D-9, outline the requirements for certain pin types of glass insulators. They have been adopted by the Edison Electric Institute and assigned the designation TD-51, 1942, and it is recommended that these specifications be accepted for publication as an A.S.T.M. tentative standard.

Revisions of Tentative Standards

Tentative Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 42 T):

This revision consists of a change in the description of the test specimen used for measuring insulation and volume resistance to refer to a particular drawing published elsewhere in the methods.

Section 34.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> 34. The test specimen shall be [either] in the form of flat plate, [or] tube, *or solid rod conforming to the dimensions shown in Fig. 2, except that the notch may be omitted. See also Fig. 2 of Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257).*

Tentative Method of Test for Saponification Number of Petroleum Products by Color-Indicator Titration (D 94 – 42 T):

This revision consists of the addition of a statement to the note indicating that the use of petroleum naphtha is not necessary when this test is applied to insulating oils.

Section 8.—Add a note at the end of the fourth sentence to read as follows:

> NOTE.—In the case of insulating oils, the addition of petroleum naphtha is not necessary.

Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 – 38 T):

This revision does not change the test method in any essential detail except to make it applicable to glass fabrics, which newer materials are now in commercial use, as well as to cloth materials.

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[1] See p. 332.

[2] These specifications were accepted as tentative by the Society and have been incorporated in the revised Tentative Specifications for Orange Shellac and Other Lacs (D 237 – 43 T), see Editorial Note, p. 258. The revised specifications are published in the 1943 Supplement to Book of A.S.T.M. Standards, Part II, p. 135; Part III, p. 229.

[3] These specifications were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 262.

Section 1.—Change to read as follows by the addition of the italicized words:

1. These methods cover procedures for the testing of varnished cloths and varnished cloth tapes, *including glass fabrics* (*Note*), to be used as electrical insulation and are directly applicable to both the "straight-cut" and "bias-cut" materials, unless otherwise stated in the method.

Add a note to read as follows:

NOTE.—Only those methods which carry a notation to that effect have been approved for testing glass fabrics.

New Section 13.—Add a new Section 13 to read as follows, renumbering the subsequent sections accordingly:

13. *Purpose.*—The purpose of this test is to determine the dielectric strength of varnished cloths and varnished cloth tapes, including glass fabrics, when subjected to a short-time or step-by-step test.

Section 31.—Renumber as Section 32 and change to read as follows by the addition of the italicized words:

32. The purpose of this test is to determine the power factor and dielectric constant of varnished cloths and *varnished cloth* tapes, *including glass fabrics*, at frequencies not exceeding 70 cycles per second.

New Section 39.—Add a new Section 39 to read as follows, renumbering the subsequent sections accordingly:

39. *Insulation Resistance.*—The insulation resistance of varnished cloths and varnished cloth tapes, including glass fabrics, shall be determined in accordance with the Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257). The test specimen shall be prepared in accordance with Fig. 1 of A.S.T.M. Methods D 257.

Section 38.—Renumber as Section 40 and change to read as follows by the addition of the italicized words:

40. The purpose of this test is to determine the effect of oil on the varnish film and dielectric strength of cloths or tapes, *including glass fabrics*.

Section 44.—Renumber as Section 46 and change to read as follows by the addition of the italicized words:

46. Specimens *of varnished cloths or varnished cloth tapes, including glass fabrics*, of sufficient size to weigh not less than 5 g. (0.18 oz.) shall be accurately weighed on an analytical balance and the weight shall be reported on some commercial unit basis such as the weight per square yard, the weight per gross yard per 1 in. in width, or the weight per roll.

Section 45.—Renumber as Section 47 and change to read as follows by the addition of the italicized words:

47. The count or threads per inch of the varnished cloth or varnished cloth tape, *including glass fabrics*, shall be determined separately on the warp and filling by counting in a space of not less than 1 in. (2.54 cm.) in at least five different places in the sample strip (or strips). The average of the five determinations shall be the count.

REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Methods of Testing Electrical Insulating Oils (D 117 – 42):

The development of synthetic insulating liquids has made it necessary to distinguish between the synthetics, known officially as "askarels," and the mineral oil type of insulation for testing purposes. Proposed Methods of Testing Askarels[4] have been prepared, therefore, it is necessary to limit the scope of the tests given in Standard Methods D 117 to the mineral oils of petroleum origin.

Also it is necessary, for certain tests, to modify the test procedures for liquids having high viscosities. This is made permissible by the addition of a note to the section on scope of these methods.

Section A of Subcommittee IV has completed several years of investigation concerning various methods of determining neutralization number of these materials. As the result of this work some changes in the methods have been recommended. Some supporting data for such changes based on round-robin tests are given earlier in this report.

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[4] See p. 353.

The committee, therefore, recommends for immediate adoption the following revisions and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Section 1.—Change to read as follows by the addition of the italicized words:

> 1. *Scope.*—These methods of testing electrical insulating oils apply to *mineral* oil *of petroleum origin* for use in transformers, oil circuit breakers, and other electrical apparatus as an insulating or cooling medium, or both.

Add a note at the end of this section to read as follows:

> NOTE.—For oils having a Saybolt Universal viscosity over 150 sec. at 100 F. (37 C.), modifications of certain test procedures are necessary. These modifications are under study and development by Committee D-9. A notation under each test procedure in question indicates where such modifications are necessary.

Section 14.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> 14. Neutralization number shall be determined in accordance with the Tentative Methods of Test for Neutralization Number of Petroleum Products [and Lubricants (A.S.T.M. Designation: D 188)] *by Color-Indicator Titration (A.S.T.M. Designation: D 663)* of the American Society for Testing Materials [using the procedure described in Section 5]. *Neutralization number may also be determined in accordance with the Tentative Method of Test for Neutralization Number of Petroleum Products by Electrometric Titration (A.S.T.M. Designation: D 664) if it is desired to use an electrometric titration method.*

TENTATIVE REVISIONS OF STANDARDS

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 42):

For some time several subcommittees of Committee D-9 and Subcommittee I of Committee D-20 on Plastics have been cooperating in the development of test methods which are generally satisfactory for all plastics as well as plastics used for insulating materials. As part of this plan of cooperation some changes are being recommended in these methods which will bring the procedures outlined by the two test methods directly in line with each other. This will also involve some slight changes in the D-20 test procedures. The following proposed revisions of this standard are therefore presented to the Society for publication as tentative:

Sections 5 to 9.—Substitute for these sections the following new Sections 5 and 6, renumbering the subsequent sections accordingly:

> 5. *Tension Tests.*—Except for conditioning as specified in the tentative revision[5] of Section 3 and for the description of test specimens as specified in Section 6, the tensile properties including both ultimate strength and modulus of elasticity shall be determined in accordance with the Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638) of the American Society for Testing Materials.
>
> 6. *Test Specimens.*—(*a*) Test specimens shall conform to the dimensions shown in Fig. 1. The surface of the specimens shall be smooth and free from scratches. Care shall be taken in machining to see that the 3-in. radius sections are not undercut. If in any specimen the width is more than 0.001 in. smaller at a junction of the straight section with the radiused shoulders than it is at the center of the straight section, such a specimen shall be discarded.
>
> (*b*) Five specimens cut lengthwise and five specimens cut crosswise of the sheet shall be tested.

Figure 1.—Change the title to read as follows by the omission of the word in brackets:

> FIG. 1.—Tension Test Specimen for [Laminated] Sheet Insulating Materials.

Change the length dimension from "about 12 in." to read "$8\frac{1}{2}$ to 12 in."

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[5] 1942 Book of A.S.T.M. Standards, Part III, pp. 1589, 1590, 1591.

Change the explanatory note to read as follows by the addition of the italicized figures and the omission of those in brackets:

When C is less than [$\frac{1}{8}$] *1/4* in., A is $\frac{1}{2}$ in., B is $\frac{3}{4}$ in., D is 0.857 in., E is 3.964 in.

When C is [$\frac{1}{8}$] *1/4* in. or over, A is 1 in., B is $1\frac{1}{2}$ in., D is 1.199 in., E is 4.648 in.

Section 14.—Change from its present form to read as follows:

14. *Apparatus.*—The apparatus shall consist of the following:

(*a*) *Testing Machine.*—Any universal testing machine of the constant rate of crosshead movement type may be used provided it is accurate to 1 per cent of the lowest load to be applied.

(*b*) *Compression Tool.*—A compression tool for applying the load to the test specimen. This tool shall be so constructed that loading is truly axial, and so that the load is applied through surfaces which are flat and parallel to the ends (or faces) of the specimen and are normal to the vertical axis of the specimen. A suitable form of compression tool is shown in Fig. 2 and consists of the following:

(*1*) *Base Plate.*—A hardened steel base plate, the upper surface of which is machined and polished to as flat a surface as nearly perfect as possible.

(*2*) *Plunger and Ball.*—A hardened steel plunger, the lower end of which is machined and polished to a flatness as nearly perfect as possible and the upper end of which is machined and polished to a hollow hemisphere to receive a polished, hardened steel ball, the diameter of which approaches that of the plunger.

(*3*) *Grinding Framework.*—A grinding framework attached to the base plate and holding the plunger so that its longitudinal axis is perpendicular to the surface of the base plate and so that its polished lower surface is parallel to the polished surface of the base plate within 0.001 in. over the entire contact area.

(*4*) *Bushing.*—A hardened steel bushing holding the plunger in the grinding framework. It shall be smooth and provided with adequate lubrication so that negligible force is required to move the plunger freely through the bushing.

(*5*) In use, the base of this compression tool shall be centered on the lower platen of the testing machine and contact with the top platen (or crosshead) shall be made between the hardened steel ball and a hardened steel plate attached to the top platen.

Fig. 2.—Add a new Fig. 2 covering a suitable form of compression tool, identical with Fig. 1 of the Tentative Method of Test for Compressive Strength of Plastics (D 695 – 42 T).[6]

Standard Methods of Testing Laminated Tubes Used in Electrical Insulation (D 348 – 42):

The following revision is intended to replace the present tentative revision[5] of Section 16 (*b*), submitted in June, 1942. It provides that a test thickness of $\frac{1}{16}$ in. shall be used instead of the $\frac{1}{8}$ in. thickness called for in the tentative revision. Experience has shown that it is necessary to make this change to prevent flashover during the test.

Section 16 (*b*).—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

(*b*) *For Testing Parallel with Laminations.*—The test specimens shall be $\frac{1}{2}$ in. in length. A hole shall be drilled into one end of the test specimen in the approximate center of the wall parallel with the major axis of the tube to a depth of [$\frac{3}{8}$] *7/16* in., leaving a thickness of [$\frac{1}{8}$] *1/16* in. to be tested. A snug-fitting metal-pin electrode, with the end ground to conform with the shape of the drill used, shall be inserted in the hole. The specimen shall be placed on a flat metal plate having a diameter at least $\frac{1}{2}$ in. greater than that of the specimen. This plate shall serve as the lower electrode. Thus, in effect the material shall be tested parallel with laminations in a point-plane gap. The diameter of the hole shall be as shown in the following table:

Nominal Wall Thicknesses of Tubes, in.	Nominal Hole Diameter for Pin Electrode, in.
[$\frac{1}{16}$ to $\frac{1}{8}$, excl.]	[$\frac{1}{32}$]
$\frac{1}{8}$ to $\frac{1}{4}$, incl.	$\frac{1}{16}$
Over $\frac{1}{4}$	$\frac{1}{8}$

NOTE.—For tubes with exceptionally high dielectric strength, [or for thin-wall tubes] surface flashover may occur. [In such cases, specimens shall be drilled to a depth of $\frac{7}{16}$ in. leaving a section $\frac{1}{16}$ in. in thickness to be tested.] *Such cases shall be noted in the report.*

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[6] 1942 Book of A.S.T.M. Standards, Part III, p. 1268.

Standard Methods of Testing Laminated Round Rods Used in Electrical Insulation (D 349 – 42):

This revision consists of a change in the tentative revision[5] of Standard D 349 and is similar to that proposed in Standard Methods D 348 – 42 given above and is recommended for the same reason.

Section 20.—Change the second sentence of the tentative revision of this section to read as follows by the addition of the italicized figures and the omission of those in brackets:

> A hole shall be drilled into one end of the test specimen in the approximate center of the rod parallel with the major axis of the rod to a depth of [$\frac{3}{8}$] *7/16* in., leaving a section [$\frac{1}{8}$] *1/16* in. in thickness to be tested.

Change the "note" following this section to read as follows by the addition of the italicized words and the omission of the words and figures in brackets:

> NOTE.—For rods with exceptionally high dielectric strength [or for small diameter rods] surface flashover may occur. [In such cases, specimens shall be drilled to a depth of $\frac{7}{16}$ in. leaving a section $\frac{1}{16}$ in. in thickness to be tested.] *Such cases shall be noted in the report.*

Standard Methods of Testing Electrical Insulating oils (D 117 – 42):

Revise the tentative revision[7] of these methods by the omission of Section 26 which reads as follows:

> 26. *Preparation of Sample.*—The sample in the container shall be shaken vigorously so as to thoroughly mix the oil and allowed to stand for 15 min. to permit the air bubbles to rise to the surface. The container shall be inverted three times before filling the test cup. The cup shall be filled with the sample to a height of not less than $\frac{3}{4}$ in. (19 mm.) above the top of the electrodes and the sample allowed to stand in the cup for 3 min. prior to the first breakdown test and 1 min. prior to the application of voltage for each succeeding breakdown.

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

Tentative Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 – 40 T):

The committee recommends that these methods be approved for reference to letter ballot of the Society for adoption as standard without revision.

Tentative Methods of Measuring Mica Stampings Used in Electronic Devices and Incandescent Lamps (D 652 – 41 T):

The committee recommends that these methods be approved for reference to letter ballot of the Society for adoption as standard without revision.

ADOPTION OF TENTATIVE REVISION OF STANDARD AS STANDARD

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 42):

The committee recommends that the tentative revision[5] of these methods, referring to conditioning to be applied to test specimens prior to testing, which have appeared for three years with minor changes, be approved for reference to letter ballot of the Society for adoption as standard.

EDITORIAL CHANGES IN TENTATIVE STANDARDS

Tentative Specifications for Phenolic Laminated Sheet for Radio Applications (D 467 – 40 T):

The following clarifying editorial change is recommended in these specifications:

Section 15 (a).—Change the word "samples" to read "the material."

Tentative Specifications for Round Phenolic Laminated Tubing for Radio Applications (D 616 – 41 T):

A similar clarifying editorial change is recommended in these specifications.

Section 17 (a).—Change the word "samples" to read "the materials."

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[7] 1942 Book of A.S.T.M. Standards, Part III, p. 1592.

APPENDIX II

PROPOSED ROUTINE METHOD OF TEST FOR GAS CONTENT OF INSULATING OIL[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. This method of test is intended for determining the gas content of electrical insulating oils of low viscosities of the general order of 100 sec. Saybolt Universal at 100 F., such as are used in capacitors and paper insulated lead-covered electric cables of the oil-filled type. The determination of gas content is desirable for any insulating oil of these properties intended for use in a degassed state. This method is intended to be used for routine tests, and shall not be used as a referee test (see Explanatory Note).

Nature of Test

2. The method consists essentially of feeding the oil into an evacuated chamber in such a manner that the oil is thoroughly exposed to the vacuum, allowing free escape of any dissolved gas. From the volume of oil admitted to the chamber, the temperature, the pressure produced, and volume occupied by the released gas, the gas volume under standard conditions of pressure and temperature may be calculated as a percentage by volume of oil.

Sampling

3. (*a*) When convenient, the degassing chamber of the measuring equipment shall be connected directly to the container from which the oil is to be sampled. This is usually not convenient and is often impossible. The method of sampling described in Paragraph (*b*), which has been in satisfactory use for many years, is recommended.

(*b*) The sample container shall consist of a stainless steel cylinder $2\frac{1}{4}$ in. in inside diameter and $9\frac{1}{2}$ in. in length, closed at the bottom. An aluminum piston shall be inserted in the bore of the cylinder accurately machined for an easy sliding fit. Two nipples, diametrically opposed, shall be inserted at the extreme bottom of the cylinder. Each nipple shall have a screw plug at the end with a gasket for sealing. All connections to the measuring equipment from the sample container should be of glass or metal tubing. Butted joints connected by short sections of heavy-walled rubber tubing may be used provided the tubing is thoroughly coated with suitable sealing compound. All samples shall be taken under slight oil pressure, with the following sequence of operations: Push the piston to the extreme bottom of the cylinder. Remove plugs from both nipples. Hold the cylinder so that the nipples point in a vertical direction. By means of a rubber tube connection, force oil in through the nipple in the lowest position and flush a few milliliters out the opposite nipple to remove any trapped air bubbles. Then insert the plug in the outlet nipple and allow oil to push the piston up to fill the cylinder. Hold the piston at the top of the cylinder with one hand and plug the inlet nipple. The aluminum piston "floats" on the oil as the level varies, due to temperature changes or removal of test samples and prevents contamination by absorption and diffusion. The piston shall be accurately fitted to move down freely with decreasing oil level to prevent voids forming under the piston which would allow rapid absorption of air by the top oil. When drawing the test sample continuously from the cylinder, it is advisable to weight the piston to insure maintenance of contact with oil. Wide variations in the results are possible in two samples from the same source unless the greatest care is taken in the sampling procedure. This phase of the test is so involved with the details of what constitutes correct practice that ability to procure consistent representative samples depends, to a great extent, on wide experience. The chief precaution to insure procurement of representative samples involves a complete flushing of all piping and hose between the sample container and sample source, such as pothead, joint, cable, oil reservoir, etc., immediately preceding collection of the sample. All long pipe lines should be eliminated where possible. After taking the sample, it is very important to see that the piston always remains in contact with the oil prior to and during withdrawal of the test specimen.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials. Published as information, June, 1943.

Apparatus

4. The apparatus, shown in Fig. 1, shall consist of the following:

(*a*) *Degassing Chamber.*—Degassing chamber, A, made of heat-resistant glass[2] (with calibrated oil well at bottom), having fixed total space volume of about 175 ml. The oil well shall have a maximum capacity of 50 ml. and shall be calibrated in 0.2-ml. divisions.

(*b*) *Stopcocks.*—Glass stopcocks, B and C, at top and bottom of chamber. Stopcock grease shall be used on all stopcocks and ground-glass joints.

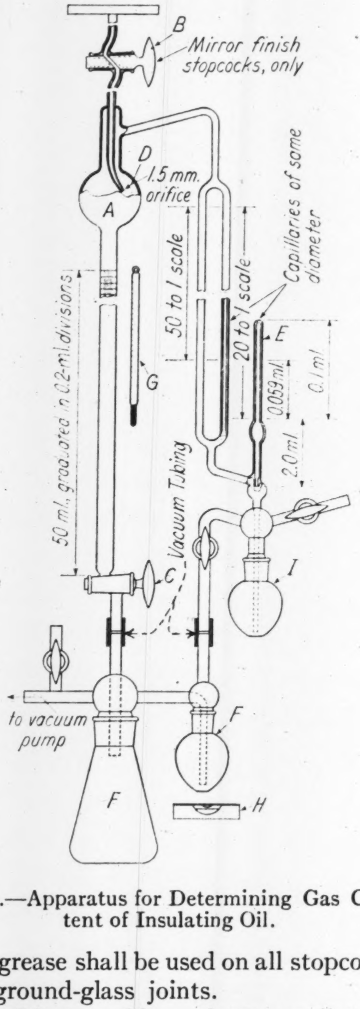

FIG. 1.—Apparatus for Determining Gas Content of Insulating Oil.

(*c*) *Pipette.*—Glass pipette or "atomizer", D, for oil in neck of chamber placed to drop oil on side of degassing chamber.

(*d*) *Pressure Gage.*—Pressure gage, E, of modified McLeod type marked for two scale factors, 50 to 1 and 20 to 1. This will provide ranges sufficient for most testing. If a higher pressure range is desired, a separate complete equipment using an absolute mercury manometer in place of the McLeod gage may be used.

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[2] Pyrex glass is satisfactory for this purpose.

(*e*) *Oil Traps.*—Two oil traps, *F*.

(*f*) *Thermometer, G.*

(*g*) *Level.*—Spirit level, *H*, normal to vertical axes of mercury columns.

(*h*) *Mercury Well, I.*

Calibration of Apparatus

5. Calibration and conditioning of the apparatus is often done by the manufacturer. If this has not been done, or if a check is desired, the following procedure may be used:

(*a*) Clean the glass assembly with a cleaning solution, wash with distilled water, and dry. Weigh the empty tester with slight film of grease on the stopcocks. Record weight in grams as *W*. Fill with distilled water and weigh. Record weight in grams as *T*. The difference between *W* and *T* is the volume of the tester in milliliters. Correction for temperature of water may be made, but is an unnecessary refinement.

(*b*) Empty and thoroughly dry equipment in oven. Pour the mercury for the McLeod gage into the mercury well, grease the stopcocks and ground-glass joints and assemble the apparatus. Attach to the mounting board (with spirit level horizontal) and align with a plumb bob and line.

Procedure

6. (*a*) Sample and apparatus should be at room temperature (20 to 30 C.).

(*b*) Evacuate the degassing chamber with stopcock *B* closed and stopcock *C* open.

NOTE.—These stopcocks must be accurately ground and kept well greased to prevent leaks.

(*c*) When the McLeod gage reading is reasonably constant and less than 0.1 mm. (absolute pressure) for all practical purposes, close stopcock *C* for about 15 min. to test for any leaks. No pressure change should be observed in this time. (Let vacuum pump run continuously to enhance seal of stopcock *C*.)

(*d*) Flush oil slowly from sample through stopcocks *B* and *C* so as to thoroughly wash walls of glass tubing and to remove any trapped air bubbles in system above stopcock *B*. (About 50 ml. of oil are commonly used for this flushing.)

(*e*) Reevacuate the degassing chamber, close stopcock *C* and by careful manipulation of stopcock *B* feed oil from the sample container into the degassing chamber at a very slow rate so that the oil falls in single drops from pipette *D*. The rate should be sufficiently slow to allow each drop to "explode," losing its entire load of gas before falling away from the pipette. This rate will generally average one drop per second or less. The oil should show no appreciable tendency to bubble or foam after falling into the oil well at the bottom of the degassing chamber.

(*f*) When 25 ml. of oil have been degassed, close stopcock *B* and read the McLeod gage and thermometer.

(*g*) Empty the oil reservoir into the oil trap, and complete the evacuation of the degassing chamber for the next test.

Calculation

7. The gas content shall be calculated as follows:

$$G = \frac{35.9\,[(V - V_1)\,P_2 - VP_1]}{(273 + t)\,V_1}$$

where:

G = gas content (at 760 mm. of mercury and 0 C.) expressed as a percentage by volume of oil,

V = known total fixed volume of apparatus in milliliters,

V_1 = volume of oil in milliliters,

P_1 = initial pressure of gas in chamber, in millimeters of mercury,

P_2 = final pressure of gas in chamber, in millimeters of mercury,

t = temperature of chamber, in degrees Centigrade.

Explanatory Note

Note.—There are several possible sources of error in this method but due to the convenience in making the test by this procedure, and the simplicity and portability of the test equipment this method is considered satisfactory for routine tests. The possible errors due to accuracy of readings are relatively small for normal ranges of gas content.

Consider the case of an oil with 0.10 per cent gas. For normal procedure, values of initial pressure and end point would be 0.100 and 0.255 mm. of mercury, respectively. This pressure increase would be developed by 25 ml. of oil tested at 25 C. (in a degassing chamber of total fixed space volume of approximately 175 ml.).

Pressures can be read to plus or minus 0.004 mm. of mercury. (This is equivalent to reading the mercury columns of the gage to plus or minus 0.20 mm. assuming a 50 to 1 scale factor of the gage). Oil volume can be read to plus or minus 0.05 ml.

Assuming errors in pressure and volume readings adding to give maximum deviation, the resultant error would be plus or minus 0.0067 in 0.10 per cent, equivalent to a percentage error of 6.7 per cent for gas contents of this low order.

Attention, however, is called to the following possible errors present in this apparatus: It is well known that the McLeod gage gives correct pressure readings only for pure gases. If vapor is present, the gage will give entirely erroneous readings, due to the high-pressure ratios present between the two columns of mercury, and consequent condensation of vapor in the high-pressure side. This would cause the pressure reading to be low with a resultant low gas content determination.

Attention should also be called to the possibility of gas leakage through any rubber tubing which may be present in the sample supply line. This may occur in spite of positive oil pressure being maintained in this line, causing, of course, higher gas content of the sample.

Also, unless sampling is done with extreme care by an experienced operator, an increase in the gas content of the sample may very easily occur, causing a high reading of gas content. Tests have also shown that all of the gas may not be entirely removed from the oil at room temperature. This error may amount to as much as 50 per cent, and, of course, results in low readings of gas content.

APPENDIX III

SIGNIFICANCE OF TESTS OF ELECTRICAL INSULATING LIQUIDS

These statements are intended to apply to the significance of tests described in the Standard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117) of the American Society for Testing Materials,[1] and the Proposed Methods of Testing Askarels.[2]

POUR POINT

Liquid dielectrics become more or less plastic solids when sufficiently cooled, due either to partial separation of wax or to congealing of the hydrocarbons or derivatives composing the product. The temperature at which the liquid just flows under the prescribed conditions is known as the "pour point."

Significance:

The pour point of a liquid dielectric gives an indication of the temperature below which it may not be possible to pour or remove the liquid from its container. It reveals the temperature at which fluidity has decreased to a point where the liquid will no longer flow freely through a restricted area.

In connection with liquid for liquid-filled cables, the pour point may be useful to indicate the point at which no free movement of liquid in the cable will take place, or to indicate the temperture at which partial separation of wax may begin.

Pour point is important in liquid dielectrics used in transformers as an index of the lowest temperature to which the material may be cooled without seriously limiting the degree of circulation of the liquid in the transformer casing.

FREE AND CORROSIVE SULFUR

If a liquid dielectric containing free and corrosive sulfur is maintained in contact with certain metals, the sulfur may attack the metal surface and form a film of the sulfide. In extreme cases this action may continue to a degree of corrosion where the strength and character of the metal are seriously impaired.

In the test for the presence of free and corrosive sulfur a polished copper strip is immersed in the liquid under test at 95 to 110 C. for a period of not less than 5 hr. The discoloring film on the copper is dissolved in a few drops of fuming nitric acid and qualitatively tested for the presence of sulfur.

Significance:

In practically all their uses, liquid dielectrics are continually in contact with copper. Since the presence of free and corrosive sulfur in the dielectric will result in deterioration of the metal to an extent dependent upon the quantity of contaminant and the time and temperature factors, the detection of undesirable forms of sulfur, even though not in terms of quantitative values, is significant as a means of recognizing the hazard involved.

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[1] 1942 Book of A.S.T.M. Standards, Part III, p. 350.

[2] See p. 353.

SPECIFIC GRAVITY

The specific gravity of a liquid is, according to the general custom of this country, the ratio of the weights of equal volumes of the liquid and water, both weights being corrected for the buoyant effect of air. The temperature limitations are indicated conveniently by the expression "Specific Gravity 60/60 F."

Significance:

Liquid dielectrics are usually sold on the basis of volumes delivered at 60 F.

In the case of mineral oils, if the specific gravity is determined at a temperature other than 60 F., volume corrections are made by the use of approved tables which show the magnitude of the correction as a function of the specific gravity of the product, regardless of its source or character.

As for the askarels, either the specific gravity must be determined at 60 F., or the proper correction for the particular material must be applied.

The specific gravity of the liquid dielectric must, therefore, be known whenever volume corrections are to be made.

In some cases, as for example, the delivery of liquid dielectric in drums, it is customary to determine weights and calculate volumes by dividing the weight of the contents of the drum by the weight per gallon. The latter figure is, of course, a function of the specific gravity of the product.

VISCOSITY

The viscosity of a fluid is the measure of its resistance to flow. In the case of liquid dielectrics an expression involving the time required for a measured volume of liquid to flow, under specified conditions, through a carefully standardized tube is known as its viscosity.

As viscosity changes rapidly with temperature, a numerical value of viscosity has no significance unless both the temperature and the type of instrument are specified. For comparative purposes it is desirable that tests should be made at standard temperatures.

In the case of mineral oils, the Standard Viscosity-Temperature Charts for Liquid Petroleum Products (A.S.T.M. Designation: D 341) of the American Society for Testing Materials[3] permits extrapolation or interpolation of viscosities at any temperature from the measured viscosities at two temperatures, with minor qualifications.

Supplemented by specific gravity data, calculation of absolute viscosities permits a more accurate comparison of materials possessing widely different densities.

Significance:

Since the rate of change of viscosity with temperature varies with different liquids, viscosity tests should, in general, be made at that standard temperature which approximates most closely the temperature of use.

In connection with a liquid dielectric for solid type cable, viscosity at a temperature approximating that of impregnation is an indication of the rate of impregnation of the paper tape. When determined at operating temperature it is a partial guide to the mobility of the liquid dielectric in the cable under performance conditions.

Viscosity is a factor in the quality of liquid dielectrics in that it reflects the ease or rate of circulation of the liquid in the transformer casing and consequently, the heat-carrying characteristics.

FLASH POINT

The flash point of a material which emits combustible vapor may be defined as the temperature to which the material must be heated in order to give off sufficient vapor to form a flammable mixture with air.

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[3] 1942 Book of A.S.T.M. Standards, Part III, p. 253.

Significance:

The flash point of a mineral insulating oil reveals the limit to which the material may be heated before the vapors emitted become a fire hazard. An unusually low flash point for a given product indicates contamination. From these standpoints the test has some value. As an indication of quality for insulating purposes it has no significance.

In the case of the askarels, the pseudo-flash which is typical of this class of products bears no relationship to the ultimate fire point or to the explosibility of the vapors or gases evolved from the liquids when heated to the boiling point. Detection of a *fire* point signifies that the dielectric is either not an askarel or is contaminated by a flammable impurity.

COLOR

The color of a liquid dielectric is determined by means of transmitted light, and is expressed by a numerical value based on comparison with a series of color standards.

Significance:

The chief significance of color as applied to a liquid dielectrics lies in the fact that it is a generally accepted index of the degree of refinement for unused liquids and an approximate measure of deterioration of products in service.

While, in general, there may be some connection between the color of a mineral insulating oil and its degree of purity and refinement, there has never been established a relationship between this characteristic and physical, chemical, or electrical stability. Therefore, color, by itself, can be considered of little inherent value.

As for the askarels, increase of color number may be indicative of the presence of impurities.

NEUTRALIZATION NUMBER

Practically all liquid dielectrics contain natural organic substances which are either weak acids or which, upon oxidation, form compounds acidic in nature. The presence of these substances is commonly referred to as "organic acidity." A properly refined product is free from mineral acids (and alkali).

"Neutralization Number" is defined as the weight in milligrams of potassium hydroxide required to neutralize 1 g. of product.

Significance:

Neutralization number is important in the examination of unused liquid dielectrics only as a check against the presence of mineral acidity or appreciable quantities of organic acidity.

In used liquid dielectrics, neutralization number may be pertinent, if compared to the value of fresh product, to detect contamination by substances with which the liquid has been in contact, or to reveal tendency toward increase in acidity due to inherent chemical change. An increase in neutralization number may be employed as a general guide for determining when a particularly well-known dielectric should be replaced by fresh material to prevent further decomposition and consequent sludging, provided suitable rejection limits have been established, and other tests confirm the need for a change.

DIELECTRIC STRENGTH

The dielectric strength of an insulating material may be defined as the minimum voltage gradient at which electrical failure or breakdown occurs under prescribed conditions. In liquids the dielectric strength is specified as the value at which breakdown takes place when the liquid is stressed between two electrodes.

Significance:

The dielectric strength of a liquid dielectric is of importance as a measure of its ability to withstand high-voltage stress without failure. It may also serve to indicate the presence of contaminating agents, such as water, dirt, or conducting particles in the liquid, one or more of which may be present simultaneously when low dielectric values are found by test. However, a high breakdown value is not a certain indication of the absence of all contaminants.

MINERAL ACIDS

The presence, or absence, of mineral acidity in liquid dielectrics is determined by shaking together specified quantities of dielectric and hot distilled water, and testing the separated water qualitatively for chlorides and sulfates.

Significance:

The significance of the test lies in its ability to reveal the presence of free mineral acid in improperly refined products and to eliminate corrosion troubles which might arise from the use of such dielectrics in service equipment.

APPENDIX IV

PROPOSED METHODS OF TESTING ASKARELS[1, 2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia 2, Pa.

Scope

1. These methods of testing apply to askarels, synthetic nonflammable type insulating liquids which, when decomposed by the electric arc, evolve only nonexplosive gaseous mixtures.

Note.—Various materials have been used successfully in taking, storing, and testing samples of askarels. Until experience has developed a strong preference, materials referred to in these methods will be understood to be approved but not specified.

Sampling

General

2. Samples from shipments of askarels shall be taken in such a manner that they will be representative of the shipment. Samples from askarels in service shall be taken in such a manner that they will represent the portion of liquid in poorest condition.

Sampling Apparatus

3. (*a*) *General.*—Sampling apparatus shall be made of materials which will have no appreciable effect upon the test results. Approved materials are glass, stainless steel, or chromium-plated metal. Sampling apparatus shall be specially constructed for consistently withdrawing samples from various types of containers or electrical apparatus.

Note 1.—The sampler shown in Fig. 1 is so constructed that it may be used in taking either a top or a bottom sample. In either case the sample is released at the bottom of the sampler.

Note 2.—The sampler shown in Fig. 2 is of the cream dipper type. Liquid enters the dipper at the top and is released into the sample bottle by opening the valve at the bottom.

Note 3.—The sampler shown in Fig. 3 is of the vacuum type. It consists of a bottle fitted with a 2-hole cork stopper, glass tubing and a syringe bulb. Liquid is drawn through the glass tubing into the bottle by means of a partial vacuum produced by operation of the bulb.

(*b*) *For Drums.*—For sampling drums the apparatus shall be so designed that liquid will enter the top of the sampler cup when the cup is submerged approximately $\frac{1}{4}$ in.

Note 4.—A convenient and simple device for sampling drums is of the cream dipper type shown in Fig. 2.

(*c*) *For Cans.*—For sampling cans of 5-gal. capacity or larger an apparatus of the dipper type, Fig. 2 may be used, or an apparatus of the vacuum type shown in Fig. 3 may be used.

(*d*) *For Tank Cars.*—For sampling tank cars the sampling apparatus may be of the type shown in Fig. 1. This is a modification of the type shown in Fig. 2 of the Standard Methods of Testing Electrical Insulating Oils (A.S.T.M.

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[1] These proposed methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

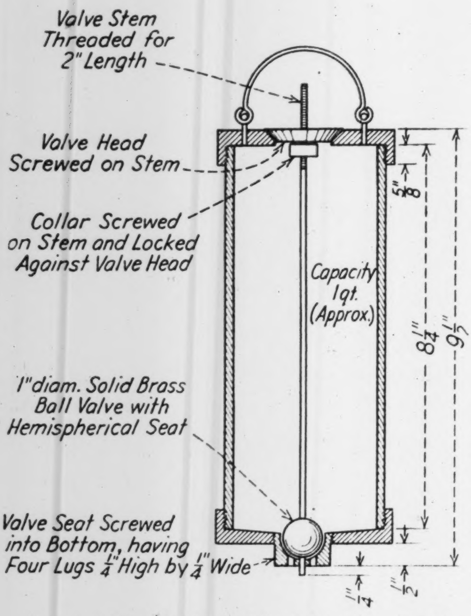
[2] Published as information, June, 1943.

Designation: D 117) of the American Society for Testing Materials.[3]

Sample Containers

4. (*a*) The following may be used as containers for the samples:

(*1*) Tin cans,
(*2*) Cork-stoppered or glass-stoppered dark glass bottles, and
(*3*) Cork-stoppered or glass-stoppered clear glass bottles in light-proof cartons.


Valve Stem Threaded for 2" Length
Valve Head Screwed on Stem
Collar Screwed on Stem and Locked Against Valve Head
Capacity 1qt. (Approx.)
1" diam. Solid Brass Ball Valve with Hemispherical Seat
Valve Seat Screwed into Bottom, having Four Lugs 1/4" High by 1/4" Wide
5/8"
8 1/4"
9 1/2"
1/4"
1/2"

NOTE.—Thief may be used for taking top or bottom sample from tank car. For bottom sample use as shown in sketch. For top sample screw valve head and collar about ¼-in. up on stem so that top valve remains fixed in open position, and submerge thief just below liquid level.

FIG. 1.—Sampler for Tank Cars.

NOTE.—The dark glass bottle has the advantage that it protects the sample from the effects of bright light. The clear-glass bottle offers the advantage that it may be examined to see that it is clean. It also allows visual inspection of the liquid before testing, permitting the detection of free water or solid impurities.

(*b*) The containers shall be clean and dry. Samples placed in glass bottles shall be kept in the dark if subsequent testing is to include color, neutralization value, power-factor or resistivity. For this reason light-proof cartons with covers shall be provided for each bottle into which the bottle with its sample of liquid shall be placed immediately after sampling. If cork stoppers are used they shall be covered with tin or aluminum foil. If glass-stoppered bottles are used, precaution shall be taken to insure that perfectly fitting stoppers are secured. A tin can fitted with a screw cap protected with a cork disk faced with tin or aluminum will endure harder usage. Tin cans shall be of the type having seams soldered on the exterior surfaces.

Cleaning of Sampling Apparatus and Sample Containers

5. (*a*) *General.*—All sampling apparatus and sample containers shall be cleaned thoroughly before using, special care being taken that no lint or other fibrous material remains in or on them. Care shall be taken to insure the complete removal of all traces of soldering flux from new containers.

NOTE.—Alcohol is a good solvent for soldering flux.

(*b*) *Sampling Apparatus.*—Sampling apparatus, before using, shall be cleaned by rinsing with dry, lead-free gasoline, Stoddard solvent, or carbon tetrachloride, and dried in the hot cabinet of the type described in Section 6 (*a*). The best procedure is to rinse it after the sampling has been finished and then place it in the hot cabinet, as in this way it will be ready for use without further cleaning when the next samples are to be taken. The rinsing material shall be used but once and then discarded.

(*c*) *Sample Containers.*—Sample containers, before using, shall be rinsed with lead-free gasoline and dried. They shall then be washed with strong soapsuds, rinsed thoroughly with distilled water and then dried by passing a current of warm air through the containers, after which they shall be immediately placed in the hot cabinet described in Section 6 (*a*). A similar washing will be sufficient for re-use of the containers that have held new liquid but those which have contained used liquid shall be washed with both lead-free gasoline and benzol before being washed with soapsuds.

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[3] 1942 Book of A.S.T.M. Standards, Part III, p. 350.

Storage of Sampling Apparatus and Sample Containers

6. (*a*) *General.*—After cleaning and until required, all sampling apparatus and sample containers shall be kept in a dry, dust free, hot cabinet in which an air temperature of approximately 60 C. shall be maintained.

(*b*) *Sampling Apparatus.*—Sampling apparatus shall be kept at all times in a vertical position in the hot cabinet in a rack having a suitable drainage receptacle at the base.

(*c*) *Sample Containers.*—Containers for samples, after being washed and dried as described in Section 5 (*c*), shall be kept unstoppered while in the hot cabinet and shall be sealed immediately on removal. During the preparation and subsequent handling of the container when introducing the sample, care shall be taken that the operator's fingers do not touch the lip of the container.

Procedure for Sampling New Askarel

7. (*a*) It is recommended that 1 qt. of liquid be taken as a sample if complete physical and chemical tests are to be made and 1 pt. if only dielectric strength tests are to be made. Sample containers should not be completely filled, about 10 per cent of the volume being left for expansion.

(*b*) Samples shall not be taken from containers until the liquid is at least as warm as the surrounding air.

(*c*) Sample containers and sampling apparatus shall be rinsed with the liquid about to be sampled.

(*d*) Drums and cans shall be preferably sampled indoors and shall be kept undisturbed for at least 12 hr. before samples of liquid are taken.

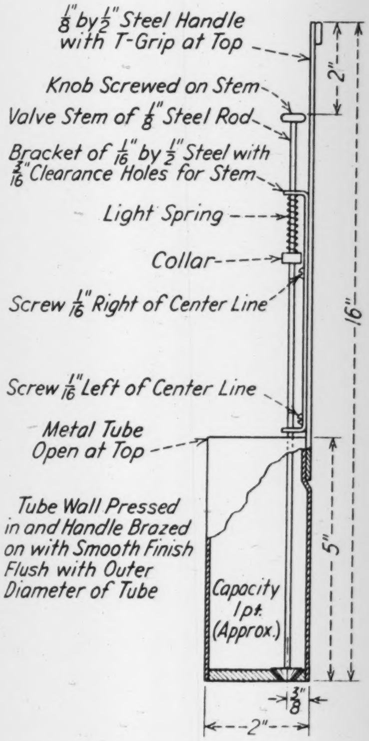
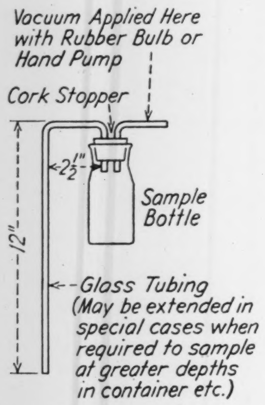

$\frac{1}{8}$" by $\frac{1}{2}$" Steel Handle with T-Grip at Top
Knob Screwed on Stem
Valve Stem of $\frac{1}{8}$" Steel Rod
Bracket of $\frac{1}{16}$" by $\frac{1}{2}$" Steel with $\frac{3}{16}$" Clearance Holes for Stem
Light Spring
Collar
Screw $\frac{1}{16}$" Right of Center Line
Screw $\frac{1}{16}$" Left of Center Line
Metal Tube Open at Top
Tube Wall Pressed in and Handle Brazed on with Smooth Finish Flush with Outer Diameter of Tube
Capacity 1 pt. (Approx.)
2"
16"
5"
$\frac{3}{8}$"
2"

Fig. 2.—Sampler for Drums.

(*e*) Drums of liquid to be sampled shall be lined up, preferably on their sides with their bungs up. The bungs shall be unsealed, removed and laid with the liquid contact sides up beside the bungholes. The dipper type sampler (Fig. 2) shall be lowered into the liquid until the lip of the cup is approximately $\frac{1}{4}$ in. below the surface of the liquid. When the sample has been transferred to the sample container, both the drum and the sample container shall be closed at once.

(*f*) When sampling containers such as cans of 5-gal. capacity or other small containers with the dipper type sampler (Fig. 2), the sampler shall be manipulated as in sampling drums or barrels. When using the vacuum type sampler (Fig. 3) the length of stem below the stopper shall be so adjusted that air bubbles are entrained as the sample is being drawn. This insures inclusion of a portion of the "surface layer." When the sample has been taken, the liquid container and the sample container shall be closed at once.


Vacuum Applied Here with Rubber Bulb or Hand Pump
Cork Stopper
$2\frac{1}{2}''$
Sample Bottle
12"
Glass Tubing (May be extended in special cases when required to sample at greater depths in container etc.)

NOTE.—In the case of samples for extremely sensitive tests such as power factor there is some evidence to indicate that brass or copper thiefs may affect the test results. The chemically clean glass thief is used to take samples for such tests from both 5-gal. cans and drums of new material. In special cases, it is used to sample apparatus in service.

FIG. 3.—Glass Sampler for Cans.

(*g*) Tank cars of liquid shall be sampled by means of a sampler of the type shown in Fig. 1. Both top and bottom samples shall be taken, as follows:

(*1*) In taking a top sample the upper valve head shall be so adjusted on the threaded shaft that it is held about ¼ in. above the top of the sampler. The sampler shall be lowered into the liquid so that the top is approximately ¼ in. below the liquid surface. The sample shall be transferred to the sample container by lifting the valve stem and so opening the lower valve.

(*2*) In taking a bottom sample the upper valve head shall be screwed down upon the collar in which position both valves remain closed until the stem is lifted. Displacement of air through the top valve largely prevents inflow of liquid through the top valve so that the sample is essentially a bottom sample. The sample container shall be stoppered immediately after it has been filled.

(*h*) When samples are being taken from different sources, precautions shall be taken to clean the sampler so as to prevent possible contamination of samples.

(*i*) Samples in either dark glass or clear glass bottles shall be stored in the dark.

Procedure for Sampling Liquids in Service

8. (*a*) It is recommended that 1 qt. of liquid be taken as a sample if complete physical and chemical tests are to be made and 1 pt. if only dielectric strength tests are to be made. Sample containers should not be completely filled, about 10 per cent of the volume being left for expansion.

(*b*) The precautions outlined in Sections 5 and 6 regarding the cleaning and storage of sampling apparatus and sample containers shall be observed as far as practicable.

NOTE 1.—As a precaution against moisture absorption, unnecessary exposure of askarel to the atmosphere should be avoided.

(*c*) Sample containers and sampling apparatus shall be rinsed with the liquid about to be sampled.

(*d*) The sampling of askarel in service is complicated by the fact that samples for electrical tests must be taken at the top surface of the liquid while samples for sediment must be taken from the bottom. Many transformers are equipped with a special sampling valve located just below the surface of the liquid. This valve terminates in a bent tube, open at the end, inside the transformer. This tube may be rotated about the horizontal center line of the valve as an axis, by a control knob on the outside of the transformer. The tube connects to an outlet on the outside of the transformer, the opening normally being capped. When not in use this tube is turned so that it points vertically upward. In this position the open end is above the surface of the liquid.

To take a sample the outside cap shall be removed and the tube rotated to a horizontal position. With the tube held in this position, just enough liquid shall be drawn off to flush the tube. This shall not be drawn into the sampling container. After the flushing, the tube shall be returned to the vertical position and the sample container substituted for the receptacle used for flushing. With the sample container held under the outlet, the tube shall then be turned until liquid just begins to flow freely through the tube. The tube shall be held in this position until a sufficient sample has been withdrawn; it shall then be returned to the vertical position.

NOTE 2.—In humid weather it is advisable to attach a metal fitting (cleaned as specified in Section 5) to extend from the valve into the sample container to prevent contamination of the sample by atmospheric moisture.

(*e*) Apparatus not equipped with special sampling valves may be sampled either by means of the dipper type sampler (Fig. 2) or the vacuum type sampler (Fig. 3). In using the vacuum type sampler, the end of the tube in the transformer shall be so adjusted that air bubbles are entrained as the sample is being drawn.

(*f*) Samples in either dark-glass or clear glass bottles shall be stored in the dark.

SPECIFIC GRAVITY

Definition

9. The specific gravity of askarels is the ratio of the weight of a given volume of the material at a temperature of 60 F. (15.56 C.) to the weight of an equal volume of distilled water at the same temperature, both weights being corrected for the bouyancy of air.

Apparatus

10. (*a*) *Hydrometer.*—A hydrometer of the floating glass type shall be used. The hydrometer shall be calibrated in terms of specific gravity. It shall be made of glass of the conventional constant mass and variable displacement type, with a scale of suitable material, clearly marked. The range may be any suitable portion of the interval between 1.0000 and 2.0000 sp. gr. The specific gravity hydrometer scale shall be divided in 0.001 of a unit of specific gravity. Each multiple of 0.01 shall be marked with a maximum length line and numbered. Each 0.005 division shall be denoted by a line longer than the line marks for the 0.001 divisions. The smallest scale subdivision shall be spaced not less than 0.75 mm. (0.03 in.) nor more than 1.5 mm. (0.06 in.). The accuracy of calibration of the hydrometer shall be such that the error at any point of the scale shall not exceed the value of one half of the smallest scale division. The scale, or an extension thereof of the hydrometer shall have plainly marked on it, the name of the manufacturer, a serial number, and the definition of the scale. There shall be a mark on the stem of the hydrometer corresponding to a fixed mark on the scale, to provide a check against possible displacement of the scale.

The hydrometer shall assume a vertical position when the instrument is floating freely in the sample. Any solid material, such as shot, paper, etc. used in the weighting of the hydrometer shall be firmly secured in place by the use of a suitable cement, the softening point of which lies well above 215 F. (101.65 C.). When mercury is used for weighting, it shall be placed in a small bulb below the main bulb of the hydrometer and completely separated from the main bulb by means of a glass partition.

NOTE.—An A.S.T.M. Gravity Thermometer of the total immersion type is suitable for use in taking the temperature in the liquid simultaneously with the gravity reading. It shall be graduated in either Centigrade or Fahrenheit degrees as specified, having a range of −5 F. to +215 F. or −20 C. to +102 C. and shall conform to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[4]

(*b*) *Hydrometer Cylinder.*—The hydrometer cylinder in which the sample for the specific gravity test is confined, shall be made of clear glass and shall be cylindrical in shape. For convenience in pouring it may have a lip on the rim. The inside diameter of the cylinder shall be at least 2.54 cm. (1.0 in.) greater than the outside diameter of the hydrometer used in it. The height of the cylinder shall be such that the length of the column of sample it contains is greater by at least 2.54 cm. (1.0 in.) than the portion of the hydrometer which is immersed beneath the surface of the sample after a state of equilibrium has been reached.

Temperature of Test

11. (*a*) The determination of the specific gravity of askarels shall be made at a temperature of 60 F. (15.56 C.) in accordance with the procedure described in Section 12 since there are no recognized tables giving the coefficients of expansion of such compounds.

(*b*) The thermometer, hydrometer, and hydrometer cylinder shall be at the same temperature as the sample to be tested. Precautions shall be taken to prevent the temperature of the sample from changing appreciably during the time necessary to complete the test. When samples having initial boiling points below 250 F. (121.1 C.) are cooled, the test cylinder shall be placed in a bath maintained at the necessary temperature during the time of test. In determining the specific gravity of askarel the temperature of the surrounding medium should not differ from the temperature of the sample by more than 5 F. (2.8 C.).

Procedure

12. (*a*) The sample to be tested for specific gravity shall be poured into the clean hydrometer cylinder without splashing, so as to avoid the formation of air bubbles. If air bubbles are formed, they shall be removed after they have collected on the surface by touching them with a piece of clean blotting paper or filter paper before the hydrometer is placed in the sample.

(*b*) The cylinder containing the prepared sample shall be placed in a vertical position in a place free from air currents. The hydrometer shall be carefully lowered into the sample to a level two smallest scale divisions below that at which it will float and shall then be released. When the hydrometer has come to rest, floating freely away from the walls of the cylinder, the specific gravity shall be read as the point at which the surface of the sample apparently cuts the hydrometer scale. In the case of samples sufficiently transparent, this point shall be determined by placing the eye slightly below the level of the liquid and slowly raising it until the surface of the sample first seen as a distorted ellipse seems to become a straight line cutting the hydrometer scale. In the case of the non-transparent sample where this procedure cannot be followed, the point on the hydrometer scale to which the sample rises above the main surface of the liquid shall be read with the eye placed slightly above the plane of the surface of the sample. This reading shall then be corrected by adding, in the case of specific gravity hydrometers, an amount equal to the height which the sample rises on the hydrometer stem above the main liquid surface. This height will vary for different liquids and different hydrometers and the amount of correction necessary will depend upon the width of the hydrometer scale graduations. The necessary correction factor shall be determined, therefore, for the particular hydrometer in use by observing the height above the main surface of the liquid to which it rises on the hydrometer scale when the hydrometer in question is immersed in a transparent liquid having a surface tension similar to that of the sample under test.

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[4] 1942 Book of A.S.T.M. Standards, Part III, p. 818.

(*c*) The temperature of the sample shall be determined from the reading of the separate thermometer placed in the sample. Although it is usually satisfactory to determine the temperature of the sample immediately after reading the hydrometer, it is recommended that in all referee tests this temperature be determined both before and after the hydrometer is read. As the specific gravity is determined at 60 F. (15.56 C.) no correction for temperature is necessary.

Reproducibility of Results

13. (*a*) Duplicate determinations shall agree within 0.0005 unit of specific gravity when the same hydrometer and thermometer are used in the tests.

(*b*) Separate laboratories using different hydrometers and thermometers should obtain duplicate results, agreeing within 0.001 unit of specific gravity. When extreme accuracy is desired, instruments certified by the National Bureau of Standards shall be used, and the necessary corrections made.

COLOR

Preparation of Reference Standards

14. (*a*) Dissolve 1.245 g. of potassium chloroplatinate (K_2PtCl_6) containing 0.5 g. of platinum, and 1 g. of crystallized cobaltous chloride ($CoCl_2 \cdot 6H_2O$) containing about 0.25 g. of cobalt in water with 100 ml. of HCl (sp. gr. 1.18). Dilute to 1 liter with distilled water. This solution has a color of 500.

(*b*) To prepare reference standards having colors of 5, 10, 15, 20, 25, 30, 35, etc., dilute 0.5, 1.0, 1.5 ml., etc., of the solution prepared as described in Paragraph (*a*) with distilled water to 50 ml. in 50-ml. tall-form Nessler tubes. Protect the reference standards from evaporation when not in use.

(*c*) The unit of color is that color produced by 1 mg. of platinum per liter.

Procedure

15. (*a*) The color of the sample shall be observed by filling a matched Nessler tube to a height equal to that in the reference standard tubes with the liquid to be examined and comparing it with the reference standards. The determination shall be made by looking vertically downward through the tubes upon a white or mirrored surface placed at such an angle that light is reflected through the columns of liquids.

(*b*) This method is good only up to a color intensity of about 100. For reading higher values the sample should be diluted with water-white benzol and the color obtained by multiplying by the dilution ratio.

Recording Colors

16. Colors between 1 and 100 shall be recorded to the nearest 5. Colors between 101 and 300 shall be recorded to the nearest 10. Colors above 300 shall be recorded in accordance with the Tentative Method of Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter (A.S.T.M. Designation: D 155) of the American Society for Testing Materials.[5]

VISCOSITY

Procedure

17. Viscosity shall be determined in accordance with either the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88)[6] or the Tentative Method of Test for Kinematic Viscosity (A.S.T.M. Designation: D 445)[7] of the American Society for Testing Materials and, unless otherwise specified, at a temperature of 37.8 C. (100 F.).

FIRE POINT

Procedure

18. Askarels as such have no fire point. As a test for contamination the lack of a fire point shall be verified in accordance with the Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92) of the American Society for Testing Materials.[8] The verification temperature shall be 200 C. (392 F.).

POUR POINT

Procedure

19. Pour point shall be determined in accordance with the Standard Method of Test for Cloud and Pour Points (A.S.T.M. Designation: D 97) of the American Society for Testing Materials.[9]

NEUTRALIZATION NUMBER

Procedure

20. Neutralization number shall be determined in accordance with the Tentative Methods of Test for Neutralization Number of Petroleum Products by Color-Indicator Titration (A.S.T.M. Designation: D 663) of the American Society for Testing Materials.[10] Neutralization number may also be determined in accordance with the Tentative Method of Test for Neutralization Number of Petroleum Products by Electrometric Titration (A.S.T.M. Designation: D 664)[11] if it is desired to use an electrometric titration method.

CHLORIDES

Nature of Test

21. This method applies to the quantitative determination of chlorides in askarels.

Extraction of Chlorides

22. (*a*) A 50-ml. sample of askarels shall be heated to 90 to 100 C. (194 to 212 F.), placed in a chemically clean 100-ml. separatory funnel and extracted with two portions of hot (90 to 95 C. (194 to 203 F.)) chloride-free distilled water. The total weight of water shall be equal to that of the 50 ml. of askarel tested. The first extraction shall be made using approximately two thirds of the water. The second extraction shall be made using the remaining one third of the water.

(*b*) Extraction shall be made by stoppering the funnel and shaking the askarel-water mixture vigorously for a minimum of 20 sec. After allowing to settle, the water extracts shall be separated into a clean centrifuge flask. The first and second extracts shall then be combined and mixed thoroughly.

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[5] 1942 Book of A.S.T.M. Standards, Part III, p. 905.

[6] *Ibid.*, p. 248.

[7] *Ibid.*, p. 983.

[8] *Ibid.*, p. 154.

[9] 1942 Book of A.S.T.M. Standards, Part III, p. 110.

[10] *Ibid.*, p. 954.

[11] *Ibid.*, p. 958.

Tyndall Beam Test for Chlorides

23. (*a*) The combined water extracts shall be centrifuged until the water shows a negative Tyndall beam effect.

(*b*) Then 0.5 ml. of a solution of silver nitrate (10 per cent), shall be added and the solution acidified with 2 drops of HNO_3 (sp. gr. 1.42).

(*c*) The test for chloride turbidity shall be made in a dark room using a spot beam of light directed across the diameter of the tube.

(*d*) The chloride content of the askarels shall be determined in parts per million by weight by comparing the chloride content of the extract with that of a standard chloride solution in the same manner.

Note.—The minimum amount of chloride that can be visually detected is 0.15 parts chloride per million parts of solution.

Arc-Formed Gases

Apparatus

24. The apparatus shall consist of the following:

(*a*) *Glass Container.*—A glass container of not less than 2-oz. capacity, fitted with suitable electrodes and outlet for gases.

(*b*) *Glass Tube.*—Glass tube filled with lightly-packed glass wool for removal of carbon particles from gases of decomposition.

(*c*) *Graduated Container.*—Graduated container for collection of gases by displacement of a suitable liquid.

(*d*) *Gas Apparatus.*—An Orsat or Burrell apparatus for analysis of the gases.

Procedure

25. (*a*) Any convenient glass vessel fitted with electrodes may be used. A suitable set-up has been made using for the sample holder a 2-oz. screw-cap type bottle fitted with a rubber stopper with two brass electrodes ($\frac{1}{8}$ to $\frac{1}{4}$-in. rod) and a glass tube outlet for the gases. Arc-formed gases shall be led (in series with the glass wool filter) to the gas collection and measuring burette. After clamping in place, the vessel shall be filled to three fourths of its capacity with the liquid to be arced.

(*b*) Electric current (110 v.d.c.) shall then be connected externally to the electrodes. The line should contain a resistance of 4 to 5 ohms. Arcing shall be carried out by manual make-and-break of the electrodes in the liquid. The external portion of the electrodes should be insulated.

(*c*) Analysis of the sample for carbon dioxide, unsaturated hydrocarbons, oxygen, carbon monoxide, saturated hydrocarbons, and hydrogen shall be carried out in the usual manner.

(*d*) The gas shall first be collected over trichlorbenzene or carbon tetrachloride which has been saturated with hydrogen chloride gas. The first absorption pipette of the gas analyzer shall then be filled with water and the halogen-containing gas determined by the percentage of gas absorbed at this stage. The residue shall then be submitted to the regular analysis for the gases previously mentioned. In order that no combustible gases may pass undetected, the explosion test for hydrogen and the saturated hydrocarbons should be carried out with oxygen.

Precautions

26. (*a*) Before collecting gases for analysis, the connecting tubing of the apparatus shall be flushed out using a

total of not less than 100 ml. of the arc-formed gases.

(*b*) Check tests shall be made on fresh samples of the liquid.

REFRACTIVE INDEX

Apparatus

27. The apparatus shall consist of the following:

(*a*) *Refractometer.*—An Abbé refractometer with scale graduated directly in terms of refractive index of the D line of sodium at a temperature of 20 C.

(*b*) *Water Supply.*—A water supply which may be varied as to temperature.

Procedure

28. (*a*) The refractometer shall be placed in front of a suitable source of light (either daylight or electric light), the thermometer inserted and circulation of water adjusted so as to bring the prisms to the desired temperature (usually 20 C.). A drop of the liquid to be tested shall then be spread upon the upper prism after which the lower prism shall be turned over and clamped. The mirror shall then be adjusted so that the light enters the telescope. The eyepiece shall be focussed on the cross-hairs and the reading lens on the scale. Upon moving the prism arm, a position can now be found where the lower part of the field is dark and the upper part light. In general, the border line will be colored and this is corrected by turning the milled head on the right of the telescope so that a sharp black and white edge is obtained. The prism arm is now moved until this black edge just crosses the intersection of the cross-hairs. The refractive index shall then be read off on the scale, the fourth decimal place being estimated.

(*b*) The accuracy of the instrument may be checked by a small test plate of known refractive index which is supplied with the refractometer. This test plate shall be attached to the upper prism with a liquid of high refractive index (usually mono-brom naphthalene). Errors may be corrected by means of a small adjusting screw on the back of the telescope.

DIELECTRIC STRENGTH

NOTE.—A method for determining dielectric strength of askarels is in preparation by the Committee.

REPORT OF SECTIONAL COMMITTEE
ON
ELECTRICAL INSULATING MATERIALS
ASA PROJECT: C59

Sectional Committee C59 on Electrical Insulating Materials held one meeting during the year: in New York, N. Y., on March 4, 1943.

The following A.S.T.M. standards, submitted to the American Standards Association on the recommendation of Sectional Committee C59, are now under consideration by the ASA looking toward their approval as American Standard:

A.S.T.M. Standard Methods of:

Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 42),
Testing Laminated Tubes Used in Electrical Insulation (D 348 - 42),
Testing Laminated Round Rods Used in Electrical Insulation (D 349 - 42),

A.S.T.M. Tentative Methods of:

Testing Molded Materials Used for Electrical Insulation (D 48 - 42 T) [Revision of American Standard C 59.1 - 1940], and
Test for Impact Resistance of Plastics and Electrical Insulating Materials (A 256 - 41 T) [Revision of American Standard C 59.11 - 1941].

In accordance with action taken at the March meeting, a ballot is now being conducted in the sectional committee on the proposed submittal of the following standards to the ASA for approval as American Standard:

NEMA Laminated Phenolic Products Standards,
NEMA Recommended Practice for Machining and Punching of Laminated Phenolic Plate, and
A.S.T.M. Methods of Testing Shellac Used for Electrical Insulation (D 411 - 42).

In view of the very limited amount of mica which is now available, it was decided to defer submittal of the NEMA Manufactured Mica Standards to the ASA for approval as American Standard until the source of supply of mica meeting the specifications is more plentiful.

Consideration of submittal of the Standard Methods for Testing Liquid Electrical Mineral Oils (D 117 - 42) is being deferred in view of forthcoming revisions.

Work on the List of Available Standards and the proposed Handbook on Electrical Insulating Materials, referred to in the 1942 report of the sectional committee, is being held in abeyance for the duration of the war.

Some preliminary work was done by the special committee giving consideration to synthetic and new materials, but it was felt that most of these of any real value are confidential and that the field is too broad for so small a group to cover. In view of the present situation, the committee was discharged and the matter tabled.

The sectional committee has expressed an interest in the activities of the War Committee on Radio—formed under the war standards procedure of the ASA at the request of the War Production Board to set up war standards for components for military radio to help speed production of radio equipment for the Army and Navy, make replacements of parts easier, and conserve materials—and has requested that any proposed

American War Standards which develop be referred to it for review so far as electrical insulating materials are concerned. If at a later date any of the war standards should be considered for approval as regular American Standards, the usual ASA procedure would be followed of having them referred to the appropriate sectional committees, and Sectional Committee C59 would no doubt be called upon in this connection.

Respectfully submitted on behalf of the sectional committee,

H. L. CURTIS,
Chairman.

R. E. HESS,
Secretary.

REPORT OF COMMITTEE D-11

ON

RUBBER PRODUCTS

Committee D-11 on Rubber Products held one meeting during the year, in Buffalo, N. Y., on March 3, 1943, in connection with the spring group meetings of A.S.T.M. committees. Several of the subcommittees have met separately during the year and considerable work has been carried on by correspondence.

Subsequent to the 1942 Annual Meeting, Committee D-11 presented to the Society through Committee E-10 on Standards proposed revisions in the Tentative Methods of Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 – 40 T). These recommendations were accepted[1] by Committee E-10 on February 18, 1943. In addition to some minor changes in the test conditions these revisions add a new procedure for determining the increase in volume by linear measurement before and after immersion, rather than by the water displacement or the specific gravity methods which were already given. This method, which is known as the Garvey method for determining volume increase, has the advantage of being adapted to rapid comparative tests of swelling when a large number of solvents or rubber compounds are involved. The revised tentative methods appear in the 1942 Book of A.S.T.M. Standards, Part III.

In the 1942 annual report of Committee D-11 mention was made of the agreement reached between Committee D-20 on Plastics and Committee D-11 as to jurisdiction over rubber-like materials which might be classified either as artificial rubbers or as plastics. Since that agreement was made it has been necessary to reopen the matter, particularly with respect to polyvinyl chloride (Koroseal) and polyisobutylene. Specifications for these materials are being prepared by Committee D-20. It has been felt that they are primarily plastics rather than rubber-like materials since they are thermoplastic and do not vulcanize. They may, however, in some cases be used in products as substitutes for rubber. Committee D-11 has therefore waived jurisdiction in favor of Committee D-20 so far as these basic materials are concerned but it reserves jurisdiction over products containing them when they are used in these products as substitutes for rubber. This question, together with the very general substitution of synthetic materials for natural rubber, has reopened the matter of revision of the scope of Committee D-11 which is now in the hands of a special committee for further study.

EMERGENCY ALTERNATE PROVISIONS AND EMERGENCY SPECIFICATIONS

During the year, Committee D-11 has utilized the emergency procedure for a number of urgent changes in the specifications for insulated wire and cable and for electrical tapes. On November 25, 1942, Committee E-10 on Standards accepted Emergency Alternate Provisions EA – D 69 and EA – D 119 in the Standard Specifications for Friction Tape (D 69 – 38) and for Rubber Insulating

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee D-11 reported the following results of the letter ballot vote of a total of 69 ballots returned from a committee membership of 123: Affirmative 53, negative 0, and 16 ballots marked "not voting."

Tape (D 119 – 38), respectively. In both cases these emergency provisions alter the composition requirements with respect to the use of new rubber and rubber hydrocarbon material in these products. Physical requirements were also changed correspondingly, as well as other details such as increasing the size of lots for sampling and eliminating the requirement on guarantee. These changes were made necessary by restrictions and scarcity of the materials formerly used in the manufacture of these products. Further war-time restrictions necessitated a change in EA – D 69 which is being submitted to Committee E-10.[2]

On January 15, 1943, Committee E-10 accepted revisions in the Emergency Alternate Provisions in the Standard Specification for Insulated Wire and Cable: Heat-Resisting Rubber Compound (EA – D 469). These revisions incorporate in the alternate provisions the initial physical property requirements of the performance rubber compound (EA – D 353), together with a heat aging requirement of 50 per cent loss from the original values after subjecting the insulation to 20 hr. exposure in the Air Pressure Heat Test (D 454 – 41). The purpose of these revisions was to conserve rubber and to provide specifications for heat resisting insulation which would comply with War Production Board rubber restrictions.

On April 15, 1943, Committee E-10 accepted revisions in Emergency Alternate Provisions EA – D 532 in the Tentative Specifications for Rubber Sheath Compound for Electrical Insulated Cords and Cables (D 532 – 39 T) and new Emergency Specifications for Chloroprene Sheath Compound for Electrical Insulated Cords and Cables (ES – 28),[2] and for Chloroprene Sheath Compound for Electrical Insulated Cords and Cables Where Extreme Abrasion Resistance is Not Required (ES – 30).[2] The changes in the Emergency Alternate Provisions with respect to the Rubber Sheath Compound (EA – D 532) were made necessary by War Production Board restrictions on the use of rubber in type WB compound. Continued and increasing difficulties had been reported in the production of satisfactory jackets because of the increasing shortage of suitable grades of rubber and reclaim until the need for immediate relief became very urgent. The new emergency specifications covering chloroprene sheaths provide for the elimination of natural rubber from this service by substitution of suitable chloroprene compounds. The substitution of this synthetic material had already been made to an increasing extent by the Navy and Signal Corps. As more chloroprene became available due to completion of new manufacturing facilities, the War Production Board gave notice that natural rubber must be eliminated from this use and that chloroprene would be allocated as a substitute. With this change imminent, the need for an industry standard covering the use became very urgent and was provided by these emergency specifications. It is probable that other synthetics will also be applied to this service in the future as they become available. The Sheath Compound ES – 28 will be used in place of that required by the Rubber Specifications D 532, and ES – 30 will take the place of the Emergency Specifications for Rubber Sheath Compound for Electrical Insulated Cords and Cables Where Extreme Abrasion Resistance is Not Required (ES – 6).

I. NEW TENTATIVE STANDARDS

Tentative Method of Test for Low Temperature Brittleness of Rubber and Rubber-Like Materials.[3]—This method has been approved by Technical Committee A on Automotive Rubber for use in evaluating the resistance to freezing temperatures of rubber and synthetics used in automotive and aeronautical products. The use of motorized equipment in extremely cold climates by the Armed Forces and the increasing altitude of flight now being attained by airplanes have created many problems involving the resistance of materials to the extreme conditions which they must withstand. These problems are, of course, new and little work has been done in the past either on the properties of materials or on the methods of testing them under these extreme conditions. No standard methods are available and the need for them at present is very great. The method here presented has been in use for some months and has proved to be a valuable tool in the selection of materials which retain flexibility at low temperatures. Research is being continued on the development of more comprehensive tests giving more precise and definite results but meanwhile it is essential that some methods be available in standard form, even though they may be altered or abandoned later as new knowledge and techniques are developed. It is with this thought in mind that Committee D-11 recommends the acceptance of this method for publication as tentative.

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[2] See Editorial Note, p. 372.

[3] These methods were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, pp. 413, and 471, respectively.

Tentative Methods of Testing Compressed Asbestos Sheet Packing.[3]—These methods have been developed by Subcommittee VI on Packings for the purpose of giving in the form of a consolidated standard a number of test procedures suitable for use in the evaluation of compressed asbestos sheet packing. Packing of this type is used for a variety of purposes but particularly as a gasketing material where severe services are encountered, such as those involving resistance to heat, heavy compression or, in the case of synthetic packing, resistance to organic solvents. The testing of the product is difficult both because of the thinness of the sheets ordinarily used and because of the nonhomogenous nature of the material which is inherently a mixture of large quantities of asbestos fiber bound together by rubber or synthetic rubber-like materials. In the past there has been little standardization of test methods as applied to the product which has resulted in a very unsatisfactory state of affairs, both to the producer and to the consumer. Large quantities of this material are now being used in aircraft engines and the need for standardized test methods has become even more urgent. Subcommittee VI has thoroughly surveyed the field and has devoted several years of study to the problem, the results of which are presented to the Society in the form of these proposed tentative methods which are recommended for publication.

Tentative Specifications for Insulated Wire and Cable: Polyvinyl Insulating Compound.[4]—Insulation compounds for electrical wires and cables made from polyvinyl chloride or from copolymers of vinyl chloride and vinyl acetate were in use to a limited extent prior to the war. In these compounds the vinyl plastics replaced natural rubber and gave properties superior for some purposes. Shortly after the outbreak of the war, all of the limited supply of these materials was reserved for the use of the Army and Navy. No standard specifications had been developed although there was a considerable background of service experience. The critical situation with respect to the supply of natural rubber now makes essential the fullest use of all available substitutes and with increased amounts of vinyl plastics becoming available through new plant construction, this material is being allocated more broadly for uses where it can replace rubber. For example, the use of rubber has been prohibited in insulation for wire and cable used on some types of ships now being constructed. Standards covering the available substitutes are therefore of the greatest importance now and are urgently needed. As a part of its program of developing these standards, Subcommittee V on Insulated Wire and Cable has prepared these Specifications for Insulated Wire and Cable: Polyvinyl Insulating Compound which are recommended for publication as tentative.

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[4] These specifications were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, pp. 434, and 416, respectively.

Tentative Specifications for Rubber and Synthetic Rubber Compounds for Automotive and Aeronautical Applications.[4]—The standardization and simplification projects on both natural and synthetic rubber compounds, which were in progress in Technical Committee A, referred to in last year's report, resulted in the publication last summer of two sets of proposed classifications in tabular form. These were given wide publicity both by the American Society for Testing Materials and by the Society of Automotive Engineers. These classifications have been recommended to consumers as a basis upon which engineers could develop product specifications as they list a range of compounds defined as to physical properties adequate for most needs. Use of these compounds in automotive and aircraft basic specifications greatly promotes simplification and standardization of rubber product specifications and thereby assures more uniform production, at the same time making possible extensive savings in material and labor and in reducing waste. These classifications were very soon adopted by several of the leading automotive manufacturers for incorporation in their specifications covering most rubber parts, except tires and tubes, and parts made from hard rubbers or sponge rubbers which were not covered by the classifications. Subsequently the Army Ordnance Department issued its Tentative Specification AXS-878 on Rubber and Synthetic Rubber for General Mechanical, Aeronautical and Automotive Application (Except Tires) which was based on these Technical Committee A classifications. Also the Naval Ordnance Department used the tables as a basis for their Ordnance Specification and Classification of Rubber Goods and Parts.

Technical Committee A through Committee D-11 now recommends that these original classifications be approved and published in the form of tentative specifications for rubber and synthetic rubber compounds and accordingly the original classifications have been rewritten as a specification. Originally the classifications were divided into two parts, the first consisting of compounds made from natural or reclaimed rubber and the second of compounds made from synthetic rubber or rubber-like materials. The first group has no particular oil-resistance characteristics required while the synthetic rubber compounds were in turn classified according to relative oil resistance. As the standardization work proceeded, the committee felt that it would be better to disregard the division according to type of rubber and to base the classification primarily on oil resistance. Under these circumstances, compounds of the first group which are designated by the prefix R may be made from any kind of rubber so long as the physical requirements are met. The same would also hold true of the second group designated as type S compounds, except that the inclusion of oil-resistance requirements restricts to a considerable extent the use of natural rubbers in these compounds. It should be understood that these specifications are general and that they do not include all of the compounds which may be needed for the almost unlimited applications where rubber and rubber-like materials are used. The committee believes, however, that they do provide a good start in standardization and that they will furnish a basis for engineers to select materials suitable for common applications in the automotive and aeronautical fields.

II. REVISION OF TENTATIVE STANDARD

The committee recommends that the Tentative Methods of Testing Sponge Rubber Products (D 552 - 41 T) be revised by changing the test period specified in Section 19 covering the procedure for compression set from "22" to read "70" hr. at 158 F. Experience has indicated that the 22-hr. period is not sufficiently long to give satisfactory evaluations of this characteristic in all cases.

III. TENTATIVE STANDARDS CONTINUED AS TENTATIVE WITHOUT CHANGE

The following seven tentative standards, under the jurisdiction of Committee D-11, are ready for submittal for adoption as standard:

Tentative Methods of:

Chemical Analysis of Rubber Products (D 297 - 41 T),
Testing Rubber Hose (D 380 - 40 T),
Test for Compression Set of Vulcanized Rubber (D 395 - 40 T),
Testing Sponge Rubber Products (D 552 - 41 T),
Testing Automotive Hydraulic Brake Hose (D 571 - 40 T),
Test for Compression-Deflection Characteristics of Vulcanized Rubber (D 575 - 40 T), and
Test for Physical State of Cure of Vulcanized Rubber (T-50 Test) (D 599 - 40 T).

As a war economy, however, the committee is withholding the recommendation for adoption of these tentative standards at this time since such action is not essential and would involve reprinting and considerable other expense. For these and other reasons all tentative standards under the jurisdiction of the committee are recommended for continuation as tentative for the time being.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 125 voting members with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials......	49	1	16
Testing Compressed Asbestos Sheet Packing..............	27	0	39
Spec. for Insulated Wire and Cable: Polyvinyl Insulating Compound.................	30	1	35
Spec. for Rubber and Synthetic Rubber Compounds for Automotive and Aeronautical Applications..................	41	2	24
II. REVISION OF TENTATIVE STANDARD			
Testing Sponge Rubber Products (D 552 - 41 T)..........	31	1	34

ACTIVITIES OF SUBCOMMITTEES

Technical Committee A on Automotive Rubber (L. A. Danse, chairman).—Although the work of Technical Committee A has centered during the year mainly on the preparation of the classifications and specifications for rubber and synthetic rubber compounds, considerable progress has been made on several other projects.

Through its Subcommittee VIII on Coolant System Hose, two specifications have been prepared and are now in process of detailed consideration. One of these specifications covers hose for use in combat tank applications where the service is severe and the hose must withstand the effect of pressure systems, various types of coolant liquids, both hot and cold temperatures, and unskilled applications to fittings. The other specification is for an all-reclaim noncombat vehicle radiator hose which is only suitable for less severe service and which should not be used except when fully approved by both producer and consumer.

Also through its Subcommittee VI on Automotive V-Belts, Technical Committee A is actively engaged in work on automotive fan belts made using synthetic rubber as well as natural rubber. An extensive test program has been carried out on belts commercially available looking toward assurance of satisfactory results from the use of synthetic materials. Considerable difficulty has been encountered in the selection of suitable laboratory tests and a field service testing program is in progress.

Subcommittee V on Insulated Wire and Cable (R. A. Schatzel, chairman).—Subcommittee V is devoting considerable attention to the effect of rubber restrictions and the use of substitute materials in insulated wire and cable as is evident by the specifications already developed covering chloroprene sheaths and polyvinyl insulation. It is anticipated that other synthetic materials will have to be used in the very near future, including GR-S rubber and probably others. The committee has decided that in writing specifications for synthetic rubber-like materials, it will be necessary to prepare separate specifications for each material as the processing characteristics and the electrical properties may vary considerably. It is anticipated that in the near future this subcommittee will present emergency specifications for insulation[2] in which the rubber constituent is entirely GR-S in one case, or mixtures of GR-S and crude rubber or reclaim in another case; also for jackets on similar bases.

Subcommittee VI on Packings (William Staniar, chairman)—This subcommittee, having completed its work on test methods for compressed asbestos sheet, is now giving consideration to test methods suitable for treated paper gasketing.

Subcommittee XV on Life Tests (H. E. Outcault, chairman)—In 1940 this subcommittee presented a Proposed Method of Test for Resistance of Rubber Compounds to Accelerated Light Aging and a Proposed Method of Calibrating a Light Source Used for Accelerating the Deterioration of Rubber. As a result of considerable study of these methods, made by the committee after they were first presented, revisions have been prepared and are now under consideration in the subcommittee. As soon as final agreement is reached, it is anticipated that these methods may be recommended for publication as tentative.[2]

This subcommittee is also somewhat concerned by the present tendency to subject rubber to accelerated aging and heat tests by the oven method at a wide variety of higher temperatures other than those approved in the Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (D 573 - 42). While it is recognized that the increasing use of synthetic materials undoubtedly makes some changes desirable, the committee feels that the temperatures actually employed should be held to a minimum number in order to make the most efficient use of existing laboratory equipment and facilities. It wishes therefore, to go on record with the recommendation that when higher aging test temperatures than 194 F. (90 C.) are desired, the temperatures used shall be confined to 212 F. (100 C.), 250 F. (121 C.), and 300 F. (149 C.). When sufficient data are available, the committee expects to take further action on a higher temperature procedure in the oven aging test.

Subcommittee XXII on Sponge Rubber (L. P. Gould, chairman).—As a result of a request from Technical Committee A, this subcommittee is working on the preparation of general specifications covering chemically blown sponge rubber, foamed latex sponge rubber, and closed cell type of sponge rubber. Proposed specifications have been drafted and circulated among interested parties for comment and suggestion. The preparation of specifications for this type of material is a particularly difficult task but they are especially needed for proper control of quality of material used by the aeronautical and automotive industries. In undertaking this work the subcommittee will render a very real service to these industries.

Subcommittee XXIV on Rubber-Coated Fabrics (A. F. Schildhauer, chairman).—This subcommittee has prepared a preliminary draft covering a group of test methods which will form the nucleus of a tentative standard on fabrics coated with rubber or rubber-like materials. It is hoped to have this ready for presentation to Committee D-11 and to the Society in the very near future.[2]

Subcommittee XXV on Low-Temperature Tests (B. S. Garvey, Jr., chairman).—Organization of this subcommittee was authorized at the meeting of Committee D-11 in June, 1942. As already mentioned, there is a great deal of interest in the low-temperature characteristics of rubbers and nonrigid plastics, and the need for adequate test procedures is acute. The subcommittee held its first meeting in March, 1943, and has already recommended the low-temperature brittleness test being submitted as a tentative standard to serve at least until more adequate methods can be developed. The subcommittee has adopted the following program for its work:

1. Four types of tests will be investigated, based on the following characteristics: (*a*) brittleness, (*b*) hardness and modulus, (*c*) loss of resilience, and (*d*) fatigue and flexing.

2. Sections will be appointed to consider each type of test. It shall be the function of each of these sections to evaluate all procedures designed to measure the specific characteristic assigned to it, to select the most suitable one, and to attempt to standardize it for general use.

The type of cold box should be specified only by performance so that each laboratory can buy or make its own.

The choice of air or liquid as the immersion medium should be made by the sections. The time required to reach temperature equilibrium should be specified.

Temperatures down to −70 F. must be considered.

3. At some stage in the development it will be necessary to consider such factors which influence low-temperature behavior as time of conditioning at the specified temperature, alternate heating and cooling, immersion in solvents or oils, etc. However, the first requisite is to develop or select adequate tests. These other problems can then be investigated.

4. The desirability of testing finished articles such as hose or packing is recognized. It is felt, however, that the best progress will be made at this time by following the procedure outlined above but the sections are advised to bear in mind the adaptability of specific tests for use with manufactured articles.

This report has been submitted to letter ballot of the committee, which consists of 125 members; 66 members returned their ballots, of whom 61 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. H. INGMANSON,
Chairman.

ARTHUR W. CARPENTER,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-11 presented to the Society through Committee E-10 on Standards a proposed revision of the Emergency Alternate Provisions in Standard Specifications for Friction Tape for General Use for Electrical Purposes (EA – D 69), which were accepted by Committee E-10 on July 20, 1943. The revised emergency provisions were published in ASTM BULLETIN, No. 123, August, 1943, p. 69, under the designation EA – D 69a, and have been issued in the form of a pink sticker for attachment to the specifications to which they apply.

On August 13, 1943, Committee E-10 accepted Emergency Specifications for Insulated Wire and Cable: Heat Resisting Synthetic Rubber Compound (ES – 33) and for Insulated Wire and Cable: Performance Synthetic Rubber Compound (ES – 34), submitted by Committee D-11. As indicated below, these emergency standards have since been accepted for publication as tentative.

Committee D-11 also submitted to Committee E-10 a revision of the Emergency Specifications for Rubber Sheath Compound for Electrical Insulated Cords and Cables Where Extreme Abrasion Resistance is Not Required (ES – 6). This recommendation was accepted by Committee E-10 on November 17, 1943, and the revised Emergency Specifications for GR-S Synthetic Rubber Sheath Compound for Electrical Insulated Cords and Cables issued under the designation ES – 6a, appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 143.

A revision of the Emergency Alternate Provisions in the Tentative Specifications for Insulated Wire and Cable: Ozone-Resistant Type Insulation (EA – D 574), recommended by Committee D-11, was accepted by Committee E-10 on November 27, 1943. The emergency provision was published in ASTM BULLETIN, No. 127, March, 1944, and has been issued in the form of a pink sticker for attachment to the specifications to which they apply.

The following recommendations have also been submitted to Committee E-10 by Committee D-11:

New Tentative Methods of:

Calibrating a Light Source Used for Accelerating the Deterioration of Rubber (D 749 – 43 T),
Testing Rubber Compounds for Resistance to Accelerated Light Aging (D 750 – 43 T),

Revision of Tentative Methods of:

Testing Asphalt Composition Battery Containers (D 639 – 41 T),
Chemical Analysis of Rubber Products (D 297 – 41 T),
Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 – 43 T), and
Testing Compressed Asbestos Sheet Packing (D 733 – 43 T).

These recommendations were accepted by Committee E-10 on November 20, 1943, and the new and revised tentative standards appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, pp. 402, 399, 463, 369, 405, and 471.

On December 27, 1943, Committee E-10 accepted the recommendation of Committee D-11 that the following specifications and methods of test be issued as tentative:

Tentative Specifications for:

Chloroprene Sheath-Compound for Electrical Insulated Cords and Cables (D 752 - 43 T) [formerly issued as Emergency Standard ES - 28],

Chloroprene Sheath Compound for Electrical Insulated Cords and Cables Where Extreme Abrasion Resistance is Not Required (D 753 - 43 T) [formerly issued as Emergency Standard ES - 30],

Insulated Wire and Cable: Heat-Resisting Synthetic Rubber Compound (D 754 - 43 T) [formerly issued as ES - 33],

Insulated Wire and Cable: Performance Synthetic Rubber Compound (D 755 - 43 T) [formerly issued as ES - 34], and

New Tentative Methods of Testing Rubber-Coated Fabrics (D 751 - 43 T).

These new tentative standards appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, pp. 449, 452, 441, 445, and 425.

REPORT OF COMMITTEE D-12

ON

SOAPS AND OTHER DETERGENTS

Due to the war and restriction on travel, Committee D-12 on Soaps and Other Detergents had decided to hold no meetings this year. However, further developments necessitated a meeting which was held on June 7 and 8, 1943, in New York, N. Y.

At this meeting, a round-table discussion was held with J. E. Simpson of the Quartermaster Corps, U. S. Army, on the subject of salt-water soaps made from synthetic detergents. Both the consumers and producers were well represented, and from the vigorous discussions, it would seem that the committee was of much assistance to the Quartermaster Corps in solving some of the problems, inherent in producing substitutes for the salt water soaps made from coconut oil.

Two papers were also presented at this meeting, one on "Synthetic Detergents as Hard Water Soaps in the War Effort" by C. J. Sunde[1] of the War Production Board, and the other on "Studies on Synthetic Detergents" by J. C. Harris of the Monsanto Chemical Co. Both papers are very interesting and instructive and will be published in the ASTM BULLETIN.[2]

The subcommittees and sections are working on numerous problems, but due to a shortage of analytical chemists in the plants, and resulting pressure on those who have not been called to war work, it is very difficult for collaborators to finish certain committee work. It is possible, however, to report progress on some activities carried on during the year.

There have been a number of changes in membership. C. C. Geddes, National Association of Dyers and Cleaners, has replaced G. P. Fulton and S. J. Walker, who have joined the Navy, and L. T. Howells of the Beach Soap Co., has rejoined the committee.

The following new members have been elected to Committee D-12 during the year:

M. Antoinette Falcone, Associated Merchandizing Corp.,
Claudius Nielsen, Mercury Chemical Co.,
Hubbel Lathrop, Western Electric Co.,
Bert Ahrens, Educational & Institutional Cooperative Service, Inc., (consulting member),
Scientific Glass Apparatus Co., represented by William Geyer,
The Emil Greiner Co., represented by J. P. Bader, and
Watson-Park Co., represented by F. A. Prisley.

Five of these new members are general interest members, restoring the committee to a proper balance between producer, consumer, and general interest members.

Walter M. Scott is now a Colonel in Chemical Warfare Service and is overseas.

Committee D-12 is cooperating with Committee B-8 on Electrodeposited Metallic Coatings and with Committee D-13 on Textile Materials.

B. S. Van Zile has transferred from Colgate-Palmolive-Peet Co. to Hercules Experiment Station, Hercules Powder Co., and Colgate-Palmolive-Peet Co. has acquired a company membership with M. F. Graham as their official representative and Messrs. M. F. Graham, E. W. Blank, Woodhead, and W. W. Sweet representing them on the various subcommittees and sections.

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[1] "Soap and Sanitary Chemicals," July, 1943.

[2] Published in ASTM BULLETIN, No. 125, December 1943, pp. 25 and 27.

Subsequent to the 1942 annual meeting, Committee D-12 recommended to Committee E-10 on Standards that the Emergency Alternate Provisions in the Standard Specifications for Chip Soap (EA – D 496) and Standard Specifications for Powdered Soap (Nonalkaline Soap Powder) (EA – D 498) be withdrawn. This recommendation was accepted by Committee E-10 on August 24, 1942.

Due to the increasing shortage of fats, the committee is planning to recommend new Emergency Alternate Provisions in the Standard Specifications for Chip Soap (D 496 – 39) and for Powdered Soap (D 498 – 39) permitting the use of 10 per cent rosin with a corresponding reduction of the titre minimum to 36.[3]

The committee has sponsored a special compilation of "A.S.T.M. Standards on Soaps and Other Detergents" issued in January, 1943, which contains all of the specifications and test methods developed by the committee. The publication also contains proposed methods for analyzing metal cleaners, which are published for discussion and criticism only, as well as an Annotated Bibliography of Aluminum Cleaning compiled by J. C. Harris, chairman of Section G on Metal Cleaners of Subcommittee II, Monsanto Chemical Co., and R. B. Mears, Metallurgical Div., Aluminum Research Labs., Aluminum Company of America. This bibliography is very timely because of the tremendously increased use of aluminum.

In order that the material would be widely disseminated and be of maximum service, the bibliography was also published in the ASTM BULLETIN in two parts in the January and March, 1943, issues.

In addition to the usual cooperative work undertaken by the various sections, Section A on Soap Methods of Subcommittee I is planning to undertake studies on methods for analyzing mixtures of soaps with other detergents. All sections reported progress and will continue cooperative studies during the coming year.

Section E on Sulfonated Detergents of Subcommittee II has been working since 1936 on a method for detergency evaluation without too much success. However, a summary of the work to date has been prepared by J. B. Crowe, the chairman of the section, and appears in an Appendix to this report. The section is continuing its study of this problem.

The committee has voted to change the names of Section B of Subcommittee I and of Section E of Subcommittee II to "Synthetic Detergents," as a more inclusive term.

Subcommittee IV on Papers and Publications has recommended that the papers already published on soaps be submitted to W. G. Morse together with the articles on caustic soda, and soda ash, prepared by members of Committee D–12. It is planned to request other members, familiar with the manufacture and properties of the other special detergents, to prepare similar articles for submission to Mr. Morse who has been asked to edit this and other material which he might have and to prepare a paper covering soaps and other detergents which would be of value to purchasers and users of these materials. This paper would then be submitted to the members of Subcommittee IV for comment, suggestions, and criticisms. When finished, this paper could be published as a paper sponsored by Committee D–12.

Last year, the committee voted to

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[3] See Editorial Note, p. 377.

request the members to pay voluntary contributions of one dollar per member to cover extra expenses of the committee. This has been submitted to the Executive Committee of the Society and was approved with the understanding that this money will be in the custody of Society Headquarters, to be disbursed on vouchers signed by the officers of the committee, and that the assessment is voluntary and should in no way be construed as a requirement of membership.

I. NEW TENTATIVE STANDARD

The committee recommends that the new proposed Method of Test for Water-Immiscible Organic Solvents Volatile With Steam in Sulfonated and Sulfated Oils,[4] prepared by Section I-B on Synthetic Detergents, be accepted for publication as tentative.

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends that the following tentative standards be revised as indicated below and continued as tentative:

Tentative Specifications for Trisodium Phosphate (**D 538 – 39 T**),[5] revised as follows:

Section 1.—Change to read as follows by the omission of the words in brackets:

> 1. These specifications [for trisodium phosphate] cover material suitable for various washing, cleaning, and scouring processes, with or without soap as conditions demand, and where a moderately strong alkaline material is desired.

Section 3.—In the table of requirements for chemical composition of trisodium phosphate, change the requirements for trisodium phosphate and phosphoric anhydride to read as follows by the addition of the italicized figures and the omission of those in brackets:

Trisodium phosphate calculated as $Na_3PO_4 \cdot 12H_2O$, from P_2O_5, min., per cent........................ [98] *97*

Phosphoric anhydride (P_2O_5), min., per cent........................[18.3] *18.1*

Section 4 (a).—Change the percentage of trisodium phosphate from "98" to "97" where it occurs in the text and in the formula.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Test for Water-Immiscible Organic Solvents Volatile With Steam in Sulfonated and Sulfated Oils..............	40	0	6
II. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Trisodium Phosphate (D 538 – 39 T)........	41	0	5
Def. of Terms Relating to Soaps and Other Detergents (D 459 – 42 T)..............	40	1	5
III. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Methods of Sampling and Chemical Analysis of Special Detergents (D 501 – 41 T)......................	41	0	5
IV. EDITORIAL CHANGE IN STANDARDS			
Spec. for Palm Oil Solid Soap (Type A, Pure; Type B, Blended) (D 535 – 41).......	41	0	5
Spec. for Palm Oil Chip Soap (Type A, Pure; Type B, Blended) (D 536 – 42)......	41	0	5
Spec. for Olive Oil Solid Soap (Type A, Pure; Type B, Blended) (D 592 – 42).......	41	0	5
Spec. for Olive Oil Chip Soap (Type A, Pure; Type B, Blended) (D 630 – 42).......	41	0	5

Tentative Definitions of Terms Relating to Soaps and Other Detergents (**D 459 – 42 T**),[6] revised by the omission of the definition for straight soap, changes in eight of the present definitions, and the addition of five new definitions.[7] Some of the revisions were made in view of comments and criticisms received from Committee E-8 on Nomenclature and Definitions on the previous definitions.

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[4] This method was accepted as tentative by the Society and appears in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 513.

[5] 1942 Book of A.S.T.M. Standards, Part III, p. 1535.

[6] *Ibid.*, p. 1544.

[7] These revised definitions were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 516.

III. ADOPTION OF TENTATIVE STANDARD AS STANDARD

The committee recommends that the Tentative Methods of Sampling and Chemical Analysis of Special Detergents (D 501 – 41 T)[8] be approved, without revision, for reference to letter ballot of the Society for adoption as standard.

IV. EDITORIAL CHANGE IN STANDARDS

Inasmuch as soaps cannot be manufactured "pure" in that they cannot be produced free from water, etc., the committee has decided to substitute the word "straight" in place of "pure" in the titles and throughout the text of the following four specifications, this change to be made editorially in these standards:

Standard Specifications for:
Palm Oil Solid Soap (Type A, Pure; Type B, Blended) (D 535 – 41),
Palm Oil Chip Soap (Type A, Pure; Type B, Blended) (D 536 – 42),
Olive Oil Solid Soap (Type A, Pure; Type B, Blended) (D 592 – 42),
Olive Oil Chip Soap (Type A, Pure; Type B, Blended) (D 630 – 42).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 74 members with the results shown in Table I.

This report has been submitted to letter ballot of the committee which consists of 74 members; 46 members have returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. P. TREVITHICK,
Chairman.

B. S. VAN ZILE,
Secretary.

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[8] 1942 Book of A.S.T.M. Standards, Part III, p. 1538.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-12 presented to the Society through Committee E-10 on Standards proposed Emergncey Alternate Provisions in the Standard Specifications for Chip Soap (D 496 – 39) and for Powdered Soap (Nonalkaline Soap Powder) (D 498 – 39). These emergency provisions were accepted by Committee E-10 on August 30, 1943, and issued under the designations EA – D 496a and EA – D 498a, respectively. They were published in ASTM BULLETIN, No. 125, December, 1943, p. 65, and have been issued in the form of pink stickers for attachment to the standards to which they apply.

APPENDIX

COOPERATIVE STUDIES ON A LABORATORY METHOD FOR EVALUATING SYNTHETIC DETERGENTS

By J. B. Crowe[1]

Synopsis

Soiled wool and cotton fabrics were used for evaluating synthetic detergents by members of Section E on Sulfonated Detergents, Subcommittee II on Specifications, of the Society's Committee D-12 on Soaps and Other Detergents. These test fabrics were washed in launderometers and graded on photometers. This paper gives a résumé of the conclusions and opinions of the cooperating members, together with brief descriptions of the methods used. Details of a typical procedure used for soiling, washing, and grading cotton and wool are appended.

With a view to obtaining information on the practicability of laboratory performance tests for evaluating synthetic detergents, Section E on Sulfonated Detergents, of Subcommittee II on Specifications, has carried out a series of tests. The following participated: G. H. Alpers, J. E. Doherty, J. C. Harris, H. L. Jones, C. L. Nutting, W. H. Tiffany, and B. S. Van Zile. This study continued over a 5-yr. period and resulted in the accumulation of 90 pages of data and pertinent information. The object of this paper is to give a résumé of the conclusions and opinions of the cooperating members, together with brief descriptions of the methods used.

Four series of launderometer tests were run. In any one series, all of the cloth was prepared in a central laboratory to minimize variations in the test fabrics. Detailed instructions were drawn up for the testing to assure that all laboratories followed the same technique. The washed fabrics (such as are used for sheeting) were graded on any type of photometer available in each laboratory, and in several instances all swatches from one series were submitted to a central laboratory for grading, after which the results were correlated with those obtained by the individual laboratories.

To aid the reader in a better understanding of the work, details of a typical procedure used for soiling and washing cotton and wool fabrics, and also for measuring detergent performance, are appended.[2]

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[1] Chairman, Section E on Sulfonated Detergents, of Subcommittee II on Specifications, of Committee D-12 on Soaps and Other Detergents; Director, Laundry and Textile Research Dept., The Procter & Gamble Co., Ivorydale, Ohio.

[2] See p. 382.

EXPERIMENTAL

Series 1—Wool Fabric Washed With Fatty Alcohol Sulfates:

Sodium lauryl sulfate and sodium oleyl sulfate (each 30 per cent active ingredient) were used as the washing agents. The fabric was a light-weight, low-count, low-twist, all-wool crepe that had first been thoroughly scoured under textile mill conditions. The soiling media consisted of carbon black, dispersed in carbon tetrachloride in which was dissolved liquid paraffin, edible olive oil, and lanolin.

Swatches of soiled fabric $2\frac{1}{2}$ by 3-in. were washed with 0.1, 0.2, and 0.4 per cent concentrations of the synthetic detergents, using

(*a*) Distilled water,
(*b*) 0.1 per cent sulfuric acid solution,
(*c*) 0.1 per cent sodium carbonate solution, or
(*d*) 0.04 per cent calcium chloride solution.

The tests were carried out in a launderometer using standard pint jars containing 200 ml. of the detergent solution, twenty $\frac{1}{4}$-in. monel metal balls, and two swatches of the soiled cloth. After a 30-min. wash at 120 F., the samples were passed through a wringer, rinsed in water at 120 F., then in cold running water, and finally air dried. The washed test pieces were first graded by whatever photometer was available in each laboratory and sent to a central laboratory so that all samples could be graded on one machine. In this work, the Zeiss-Pulfrich, the Lange photoelectric and Martens photometer, and the Hess-Ives tintometer were used.

Discussion of Results.—There was considerable variation in the results from the different laboratories. In fact, even when the fabrics were graded in one central laboratory on one machine, wide variations still existed. It was observed that the soiled cloth did not wash evenly, especially where a large percentage of the soil was removed. This made it difficult to get an accurate whiteness grade on the washed fabric. The fabric was so light in weight and so loosely woven that it frayed easily from the action of the monel balls and shrunk considerably. It was also observed that the cloth tended to roll and remain in a ball throughout the wash, thus undoubtedly contributing to the uneven washing.

Series 2—Cotton Fabric Washed With Fatty Alcohol Sulfates:

The detergents and conditions of testing were identical with those used in Series 1, with the exception that soiled cotton was used in place of soiled wool. The fabric was Indian Head 54 in. wide, permanent finish, made by the Nashua Manufacturing Co. The cloth was desized and then soiled in a dispersion of "Oil Dag Concentrated" and Wesson Oil in carbon tetrachloride.

More explicit washing instructions were given than for Series 1, hoping to minimize the inconsistencies shown by the previous test.

Discussion of Results.—The results obtained with the soiled cotton were in closer agreement than shown by Series 1 with wool. However, there was still considerable disagreement between laboratories as exemplified by the fact that some reported sodium lauryl sulfate superior to sodium oleyl sulfate, some the reverse, while a third group could see no difference.

A disturbing fact was the report by some laboratories that distilled water alone removed more dirt than the highest concentration (0.4 per cent) used of both detergents.

Series 3—Cotton Fabric Washed with Fatty Alcohol Sulfates:

The same type of soiled cotton and washing technique used in Series 2 was employed in this study. The washing instructions emphasized that the tests should be run within a predetermined time to minimize variations in the effect of aging on the ease of removal of the standard soil.

The results paralleled those obtained in Series 2.

Series 4—Wool and Cotton Fabrics Washed with Oleic Amide of the Sodium Salt of Methyl Taurine and also with Alkyl Aryl Sulfonate:

The two detergents were: the oleic amide of the sodium salt of methyl taurine (35 per cent active ingredient), and sodium alkyl benzene sulfonate (30 per cent active ingredient). The detailed instructions for soiling, washing, and grading are appended.[2]

The washed pieces were graded in each laboratory and subsequently in a central laboratory using a Lange photometer. In order to eliminate differences in grading procedure between laboratories, the washed fabrics were graded at both ends and in the center on each side, giving a total of six measurements for each fabric. In order to make the surface characteristics of all samples alike, the fabrics were ironed smooth before whiteness readings were made.

Discussion of Results.—The soil from cotton pieces washed out quite uniformly; the wool samples, however, were mottled in some cases after washing.

There were decided variations in results obtained by the individual laboratories. When graded in the central laboratory, the differences were considerably minimized although unfortunately large discrepancies still existed.

CONCLUSIONS FROM COOPERATIVE TESTS

The reproducibility of results between different laboratories using soiled cloth for evaluating synthetic detergents left much to be desired. Some laboratories reported difficulty in obtaining check results even in their own individual tests, in spite of having personnel trained in running launderometer detergency work. For this reason, this method should not be considered sufficiently accurate for referee work on synthetic detergents. However, the procedure might be considered quite useful for control work within an organization, particularly if the detergent application has been correlated with the use of soiled cloth in the launderometer.

If used as a control tool by a laboratory, great emphasis should be placed on the necessity for the personnel to follow exactly the same procedure in every test. Time of washing, temperature, concentration and volume of solutions, number and type of balls used, speed of the launderometer, method of soiling, and age of soiled fabrics are factors having a decided bearing on the results.

GENERAL DISCUSSION

Previous History of Soiled Cloth Studies:

Some years ago, extensive tests were conducted by the Detergents Committee of the American Oil Chemists Society to evaluate the detergency of soap (not synthetic detergents) using soiled cloth in launderometers. After considerable work, this committee abandoned the project feeling there was "no possibility of regarding it (the soiled cloth—launderometer method) as a general method of evaluating detergents," .... although "useful for making comparisons under limited conditions."[3] Since that work, there may have been some improvements in the manner of applying the soil to the cloth, but there have been no radical changes except to include soiled wool as well as cotton.

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[3] Oil and Soap, Vol. 10, p. 232 (1933).

Synthetic Detergents versus Soap:

Determining the detergency of synthetic detergents is much more complex than with soap. In the case of soap, the products are always used either as neutral soap or built with alkali. With these newer synthetic detergents, however, the products can be used in almost any degree of alkalinity or acidity, with or without neutral salts, with various organic agents, etc. This complicates the problem, for a detergent may appear good when used in alkaline solution, but be poor in acid. In contrast, another synthetic detergent may show the opposite behavior.

Relation of Soiled Fabric to Dirt Load Encountered in Practice:

Soiled fabrics are prepared by more or less "standard" methods, generally by impregnation with a mixture of oily material and some type of carbon. In contrast, the dirt load encountered in practice varies widely in amount, composition and also the way it gets on or into the material. The practical dirt load can vary from a mixture of lime and grease found on sheeps wool to ground-in carbon dirt on a soiled shirt. It can vary from a pigment-pitch blend used on optical lenses to an acid-inert dirt mix found on pickled metal. Even in such a limited field as the lubrication of textile fibers, the oil used for spinning varies from saponifiable to unsaponifiable oils and mixtures. Obviously, no one or even two soiled fabrics can duplicate the variety of soils found in practice, all of which are removed with greater or lesser ease during the scouring or washing process.

The Effect of Agitation on Dirt Removal:

Type of agitation and mechanical action greatly affect the relative detergency of cleaning agents. No one standardized method of agitation, such as is obtained in a launderometer, can hope to duplicate them all. Differences between such means of cleaning as squeeze roll action in washers, revolving cylinders, rotating brushes, and soaking and hand scrubbing are great. Each process has a distinct bearing on the ease or difficulty with which soil is removed from the surface to be cleaned.

Material Being Cleaned:

Any one or two types of soiled fabrics can at best correspond to only a small percentage of materials to be cleaned. In textiles alone, wool, cotton, silk, and the various synthetic fibers attract, hold, and release soil to a different degree. In addition, such factors as the twist of the yarn and fabric construction further complicate the problem. This is just one small field in the over-all cleaning problem.

GENERAL CONCLUSIONS

Many laboratory evaluations using soiled fabrics in launderometers have shown certain products or combination of products to be outstandingly good or bad. Under actual operating conditions in textile mills and laundry plants, completely different results have often been obtained. For example, soiled wool usually shows alkali to be inert or harmful to dirt removal when used with certain synthetic detergents. Actually, in practically every case, alkali is found to be helpful under practical conditions of usage.

In view of this, the best method to date of evaluating synthetic detergents for a specific application is under the actual operating conditions where they are to be used.

TYPICAL PROCEDURE USED FOR SOILING AND WASHING COTTON AND WOOL

Cotton:

1. *Fabric Used.*—Indian Head, 54 in. wide, permanent finish, made by Nashua Mfg. Co.

2. *Soiling Formula.*—The soiling formula used was 6 g. Oildag (Acheson Colloids Co., Port Huron, Mich.), 1.7 g. Wesson oil, and 3000 ml. carbon tetrachloride.

3. *Soiling Procedure.*—Cotton strips 5¼ in. wide by 10 yd. long are washed for 20 min. in 0.25 per cent tallow soap (88 per cent real soap) at 180 F., thoroughly rinsed in distilled water, and ironed. These strips are soiled in a machine after which they are dried immediately. The strips are given 3 passes through the standard soil solution which makes them approximately 55 per cent black when tested on the Lange Reflectometer.

Wool:

1. *Fabric Used.*—Botany worsted, style No. 404.

2. *Soiling Formula.*—The soiling formula used was 0.5 g. lampblack (grinders No. 2 from Bihn & Wolf Co., Phila.), 7.5 g. edible tallow, 25.0 g. Nujol, and 4000 ml. carbon tetrachloride.

3. *Soiling Procedure.*—A strip of wool cloth 9 in. wide and 21 ft. long is soiled mechanically. The cloth is passed through the soiling solution and then through weighted squeeze rolls. The soiling is continued for 10 passes through the machine which requires 16.5 min. The cloth is then removed from the machine and dried for 1 hr. in a yarn dryer at 85 C.

General Washing Technique:

The tests were made in duplicate using both soiled wool and soiled cotton fabrics in water containing 50 ppm. and 300 ppm. hardness as shown in Table I.

TABLE I.

Test	Detergent	Concentration, per cent	
No. 1	None	None (water of either 50 or 300 ppm. hardness will be used)	
2	Sodium carbonate	0.25	
3	The standard detergent *A*	0.25	
4	Detergent under consideration *B*	0.25	
5	Detergent *A* + sodium carbonate	0.25	0.20 detergent *A* 0.05 sodium carbonate
6	Detergent *B* + sodium carbonate	0.25	0.20 detergent *B* 0.05 sodium carbonate

The test pieces washed in water containing 50 ppm. hardness were marked with an *S;* the 300 ppm. water test pieces were marked with an *H*. Since four washes of 10-min. duration each were made, the tags were marked accordingly. For example, detergent *B* in 50 ppm. water would bear the designation 4S1, 4S2, 4S3, and 4S4. The initial number 4 in each designation refers to the test designation in the above table, *S* refers to 50 ppm. water, and the 1, 2, 3, or 4 refer, respectively, to the first, second, third, or fourth 10-min. washing intervals. In 300 ppm. water this test would be marked 4H1, 4H2, 4H3, and 4H4.

Test Procedure:

Reagents.—

1. Weigh out 17.6200 g. of calcium chloride ($CaCl_2 \cdot 2H_2O$) and dissolve in approximately 200 ml. of distilled water.

2. Weigh out 19.7200 g. of magnesium sulfate ($MgSO_4 \cdot 7H_2O$) and dissolve in approximately 200 ml. of distilled water.

3. When both salts have dissolved, make each up separately to 1 liter with distilled water.

4. Mix the solutions.

5. Add 8 liters of distilled water and thoroughly mix the resulting product.

6. For water of 50 ppm. hardness dilute 25 ml. of solution from step 5, to 1 liter with distilled water.

7. For water of 300 ppm. hardness dilute 150 ml. of solution from step 5 to 1 liter with distilled water.

NOTE.—Distilled water unless freshly boiled will generally have a pH of approximately 5.5 to 6.4. The addition of the salts of steps 1 and 2 to the unbuffered distilled water will buffer the pH at the desired point. *Do not* adjust the pH of the water with alkali or acid. Use freshly distilled water if necessary.

The amounts of salts added in steps 1 and 2 produce a water of 2000 ppm. hardness, as ppm. $CaCO_3$, 60 per cent of which is attributable to calcium and 40 per cent to magnesium.

8. The sodium carbonate used shall be c.p. anhydrous grade.

9. Samples of detergents *A* and *B* were furnished and used as "received."

Wash Test—Cotton Fabric:

1. Make the wash tests in duplicate in both 50 ppm. and 300 ppm. water.

2. Detergents and their concentrations shall be as follows:

Detergent	Concentration, per cent
None (use water of required hardness)	0
Sodium carbonate	0.25
Detergent *A*	0.25
Detergent *B*	0.25
Detergent *A* + sodium carbonate	0.25 { 0.20 detergent *A* 0.05 sodium carbonate
Detergent *B* + sodium carbonate	0.25 { 0.20 detergent *B* 0.05 sodium carbonate

3. Weigh 2.500 g. of sodium carbonate into a beaker, and make up to 1 liter with 50 ppm. water or 300 ppm. water, whichever is under test.

Note.—Weigh these quantities on an accurate balance.

4. Repeat step 3 with detergents *A* and *B*, respectively.

5. Weigh 2.000 g. of detergent *A* and 0.500 g. sodium carbonate into a beaker, and make up to 1 liter with 50 ppm. or 300 ppm. water, whichever is under test.

6. Repeat step 5 with detergent *B* and sodium carbonate.

7. Adjust the temperature of the Launderometer to 140 ± 2 F.

8. Adjust the Launderometer speed *when loaded* to 42 rpm.

9. Place 10 rubber balls in each pint container.

10. Preheat the solutions indicated in step 2 to 173 ± 3 F.

11. Measure 100 ml. of solution into each of the jars of the duplicate test.

12. Place the properly marked swatches in the jars (one swatch to a jar).

13. Cap and place in Launderometer and wash for exactly 10 min.

14. Remove and drain the jar, discarding the used solution and squeezing excess solution from the duplicate swatches.

15. Cut off a $1\frac{1}{2}$-in. strip from each of the duplicate swatches.

16. Twice rinse the two $1\frac{1}{2}$-in. strips of the duplicate determinations in water of the hardness in question. The rinses shall be two, of 100 ml. each at 100 F. and the strips shall be squeezed five times in each beaker of rinse water. The strips are then air dried.

17. Repeat steps 9 through 16 with the remaining portion of the original swatch until four 10-min. washes have been made.

Wash Test—Woolen Fabric:

1. The wash tests shall be made in duplicate in both 50 ppm. and 300 ppm. water.

2. Detergents and their concentrations shall be as follows:

Detergent	Concentration, per cent
None (use water of required hardness)	0
Sodium carbonate	0.25
Detergent *A*	0.25
Detergent *B*	0.25
Detergent *A* + sodium carbonate	0.25 { 0.20 detergent *A* 0.05 sodium carbonate
Detergent *B* + sodium carbonate	0.25 { 0.20 detergent *B* 0.05 sodium carbonate

3. Weigh 2.500 g. of sodium carbonate into a beaker, and make up to 1 liter with 50 ppm. water or 300 ppm. water, whichever is under test.

Note.—Weigh these quantities on an accurate balance.

4. Repeat step 3 with detergent *A* and *B*.

5. Weigh 2.000 g. of detergent *A* and 0.500 g. of sodium carbonate into a beaker, and make up to 1 liter with 50 ppm. or 300 ppm. water, whichever is under test.

6. Repeat step 5 with detergent *B* and sodium carbonate.

7. Adjust the temperature of the Launderometer to 110 ± 2 F.

8. Adjust the Launderometer speed when loaded to 42 ± 2 rpm.

9. Place 10 rubber balls in each pint container.

10. Preheat the solutions indicated in step 2 to 143 ± 3 F.

11. Measure 100 ml. of solution into each of the jars of the duplicate test.

12. Place the properly marked swatches in the jars (one swatch to a jar).

13. Cap and place in Launderometer and wash for exactly 10 min.

14. Remove and drain the jar discarding the used solution, squeezing excess solution from the duplicate swatches.

15. Cut off a 1½ to 2-in. strip from each of the duplicate swatches.

16. Twice rinse the two 1½ in. strips of the duplicate determinations in water of the hardness in question. The rinses shall be two, of 100 ml. each, at 100 F. and the strips shall be squeezed five times in each beaker of rinse water. The strips are then air dried.

17. Repeat steps 9 through 16 with the remaining portion of the original swatch until four 10-min. washes have been made.

Evaluation of Swatches:

Read the washed, dried swatches on any photometer available.

REPORT OF COMMITTEE D-13

ON

TEXTILE MATERIALS

Committee D-13 on Textile Materials held two meetings during the year in New York, N. Y.: one on October 14 and 15, 1942, and the other on March 10 to 12, 1943. The number of members and guests registered was 123 and 180, respectively. The March meeting was the largest in the history of the Committee. Of the 28 subcommittees or sections which compose the organization of Committee D-13, 17 held sessions at the fall meeting and 16 at the spring meeting. The executive direction of the committee's affairs required one meeting of the officers and two meetings of the Advisory Committee.

When the above facts, together with the numerous subcommittee activities and the many recommendations regarding standards reported herein, are considered in the light of the conditions imposed by the war, it is felt that interest in standardization in the textile field has been well maintained. The officers of the committee fully realize the difficulties caused by shortages of personnel and time, and take this opportunity to express their sincere appreciation for the cooperative and valuable service rendered by the chairman and members of the working organization during the past year.

The Advisory Committee has approved of the following changes in subcommittee and section chairmen: M. E. Campbell as chairman of Subcommittee A-1, Section I on Cotton *vice* R. W. Webb; O. G. Edwards as chairman of Subcommittee A-1, Section II on Cotton Yarns and Threads *vice* E. O. DeFore *vice* A. C. Clifford; V. W. Lenz as chairman of Subcommittee A-8, Section IV on Chemical Tests *vice* A. L. Simison; O. P. Beckwith as chairman of Subcommittee B-5 on Sampling, Presentation and Interpretation of Data *vice* A. G. Ashcroft; D. C. Scott as chairman of Subcommittee C-3 on Membership *vice* G. E. Hopkins.

Society appointments of representatives from the membership of Committee D-13 were as follows: K. H. Barnard on the Sectional Committee on Fastness of Colored Textiles (ASA Project: L14) for the duration of the war *vice* W. M. Scott; A. G. Ashcroft to the Advisory Committee on Ultimate Consumer Goods of the ASA *vice* H. J. Ball; A. J. Kellner on the Inter-Society Color Council *vice* C. J. Huber.

For several years various special committees have been functioning on matters not dealing directly with standardization work. These have now been incorporated into the organization of Committee D-13 by the establishment of a new "C" classification of subcommittees as follows: C-1, Officers Committee; C-2 on Papers; C-3 on Membership; C-4 on Publicity.

The present membership of Committee D-13 is 260; of whom 116 are classified as producers, 50 as consumers, and 94 as general interest members.

RECOMMENDATIONS AFFECTING STANDARDS

Committee D-13 is presenting for publication as tentative four new methods of test and definitions for textile materials, and is recommending the revision of one tentative standard, the revision of one standard for immediate adoption, tentative revisions in four standards, and the adoption as standard of four tentative standards. The revisions being presented are included in detail in the Appendix and are accompanied, where necessary, by brief statements of the reasons for such revisions. The new tentative standards and the existing standards affected are listed in Table I and are also referred to under subcommittee activities.

These recommendations have been submitted to letter ballot of the committee which consists of 260 members, the results being shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

The recommendations being submitted indicate that the various subcommittees and sections have been quite active during the year. Many important projects are under consideration and are mentioned briefly in the following summary of subcommittee activities.

Subcommittee A-1 on Cotton and Its Products (K. B. Cook, chairman):

Section I on Cotton (R. W. Webb, chairman) is recommending that the Tentative General Methods of Testing Cotton Fibers (D 414 – 40 T) be continued as tentative. No criticism of the methods has been received and it is felt that they are as good as can be offered at present, but the committee feels that they should be given further review.

Section IV on Tire Fabrics (Martin Castricum, chairman) recommends for publication as tentative a revision of the Standard Methods of Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 – 42) which adds the constant specimen-rate-of-load tester as an alternate to the pendulum type tester for determining strength of tire cord, as given in the Appendix.

The section has under consideration the reporting of strength and elongation results on rayon tire cord on a specified regain basis; a study of types of jaws used in cord testing machines with particular reference to elongation tests; thickness measurements on tire cords;

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Testing and Tolerances for Rope (Leaf and Bast Fibers)	29	1	66
Testing and Tolerances for Spun, Twisted, or Braided Products Made from Flax, Hemp, Ramie, or Mixtures of These Fibers	29	1	66
Test for Air Permeability of Textile Fabrics	56	2	38
Def. of Terms Relating to Textiles (D 123 – 43 T)	77	0	19
Test for Commercial Weight of Rayon Staple (D 540 – 43 T)	36	0	60
II. REVISION OF TENTATIVE STANDARD			
Methods for Identification of Fibers in Textiles (D 276 – 41 T)	71	2	23
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Methods of Testing and Tolerances for Yarns Spun from Wool Mixed with Fibers Other than Wool (D 508 – 39)	44	0	52
IV. TENTATIVE REVISIONS OF STANDARDS			
Methods of Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 – 42)	35	0	61
Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 – 42)	45	0	51
Methods of Testing and Tolerances for Spun Rayon Yarns and Threads (D 507 – 42)	39	1	56
Spec. and Methods of Test for Asbestos Tape for Electrical Purposes (D 315 – 41)	25	0	71
V. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Method of Test for Hard Scoured Wool in Wool in the Grease (Laboratory Scale Operations) (D 584 – 41 T)	27	0	69
Methods of Testing and Tolerances for Woven Glass Tubular Sleeving and Braids (D 581 – 40 T)	21	0	75
Methods of Testing and Tolerances for Woven Glass Fabrics (D 579 – 41 T), as revised	23	0	73
Methods of Testing and Tolerances for Woven Glass Tapes (D580 – 41 T), as revised	23	0	73

rayon cord fatigue testing; method of calibration of pendulum type testers for elongation; and a method for the determination of yards per pound of tire cord.

Section V on Heavy Cotton Woven Fabrics (B. L. Whittier, chairman) is studying the feasibility of establishing standard regains for heavy ducks and length of time required to establish moisture equilibrium in heavy ducks. Action will be taken on specifications for numbered ducks as soon as Federal Specifications, now in process of revision, are issued.

Subcommittee A-2 on Rayon and Its Products (A. M. Tenney, chairman):

Section I on Rayon (J. B. Goldberg, chairman) recommends for publication as tentative a revision of the Standard Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 – 42) to specify distance between clamps for both type A and type B testing machines, as given in the Appendix.

Section III on Rayon Staple and Spun Rayon Yarn (K. B. Cook, chairman) recommends for publication as tentative a revision of the Standard Methods of Testing and Tolerances for Spun Rayon Yarns and Threads (D 507 – 42) to include appearance standards for spun rayon yarns, as given in the Appendix.

The section is recommending for publication as a tentative revision of the Standard Methods of Testing Rayon Staple (D 540 – 42) a new Method of Test for Commercial Weight of Rayon Staple.[1]

Subcommittee A-3 on Wool and Its Products (A. G. Ashcroft, chairman):

Section I on Wool (Werner von Bergen, chairman) recommends the adoption as standard of the Tentative Method of Test for Hard Scoured Wool in Wool in the Grease (Laboratory Scale Operations) (D 584 – 41 T). No adverse criticism of this method has been received.

The committee is actively engaged in establishing standards for wools below grade 50's and in studying new methods of test.

Section III on Woolen and Worsted Yarns (Fred Noechel, chairman) recommends for immediate adoption a revision of the Standard Methods of Testing and Tolerances for Yarns Spun from Wool Mixed with Fibers Other Than Wool (D 508 – 39) which increases its applicability to yarns spun from any mixture of fibers of different types, as given in the Appendix.

Section IV on Pile Floor Covering (J. N. Dow, chairman) is studying problems which have developed particular importance because of war conditions such as methods for identification of fibers other than wool in floor coverings, and methods for determining the susceptibility of pile floor coverings to matting.

The section is preparing a program for post-war research on the correlation of laboratory wear testers with actual service tests for fabrics containing vegetable and synthetic fibers in the pile.

Subcommittee A-4 on Asbestos and Its Textile Products (F. S. Mapes, chairman):

This subcommittee is recommending for publication as tentative a revision of the Standard Specifications and Methods of Test for Asbestos Tape for Electrical Purposes (D 315 – 41) which specifies the dead weight dial micrometer method for measurement of thickness, as given in the Appendix.

The subcommittee recommends that the Tentative Methods of Testing Asbestos Tubular Sleeving (D 628 – 41 T) be continued as tentative pending a proposed revision of the wall thickness tolerances.

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[1] These methods were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, pp. 509, 501, 497, and 494, respectively.

Owing to present conditions, the subcommittee finds that active work on its high-temperature tests on asbestos cloths must be confined to developing a method and apparatus which could be used at a future date.

Subcommittee A-5 on Bast and Leaf Fibers and Their Products (A. R. Howe, chairman; H. J. Hayes, acting chairman):

This subcommittee is recommending for publication two new tentative standards: namely, proposed Tentative Methods of Testing and Tolerances for Spun, Twisted, or Braided Products made from Flax, Hemp, Ramie, or Mixtures Thereof,[1] and Methods of Testing and Tolerances for Rope (Leaf and Bast Fibers).[1]

The subcommittee has developed a classification of bast and leaf fibers which has been submitted to Subcommittee B-2 on Definitions for inclusion in the Glossary of Terms Relating to Textile Materials, published in the compilation of A.S.T.M. Standards on Textile Materials.

Subcommittee A-6 on Household and Garment Fabrics (Ruth O'Brien, chairman):

This subcommittee recommends that the Tentative Specifications for Bleached Wide Cotton Sheeting (D 503 - 40 T); All Wool, All Cotton, and Wool and Cotton Blanketing (Household) (D 576 - 40 T); Bleached Cotton Broadcloth (D 504 - 41 T); and Medium-Weight Cotton Corduroy Fabrics (D 625 - 41 T) be continued as tentative because of the abnormal situation regarding the qualities of civilian textiles due to the curtailed and fluctuating status of textile production for civilian use as a result of Federal regulations and of the inability of textile laboratories to do any appreciable amount of research on civilian goods in addition to urgent war work on hand.

Subcommittee A-8 on Glass Fiber and Its Products (Dean Harvey, chairman):

This subcommittee recommends the adoption as standard of the Tentative Methods of Testing and Tolerances for Woven Glass Tubular Sleeving and Braids (D 581 - 40 T).

The subcommittee also recommends the adoption as standard of the following tentative standards, revised as given in the Appendix: Tentative Methods of Testing and Tolerances for Woven Glass Fabrics (D 579 - 41 T) and Tentative Methods of Testing and Tolerances for Woven Glass Tapes (D 580 - 41 T).

The subcommittee is recommending that the Tentative Methods of Testing and Tolerances for Glass Yarn (D 578 - 40 T) be continued as tentative pending the inclusion of yardage as well as construction in the designation of the yarn, and that the methods be editorially revised as given in the Appendix.

Subcommittee B-1 on Methods and Machines (W. D. Appel, chairman):

Section I on Methods (W. D. Appel, chairman) recommends for publication as tentative a new proposed Tentative Method of Test for Air Permeability of Textile Fabrics.[1]

This section is recommending that the Tentative Methods for Identification of Fibers in Textiles (D 276 - 41 T) be revised as appended hereto.[2]

Section II on Machines (H. J. Ball, chairman) is giving particular attention to methods of calibrating pendulum type testers and the allowable error for textile testing machines.

Section III on Atmospheric Conditions (R. H. Brown, chairman) is developing a method for determining moisture in textiles by oven drying.

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[2] These revised methods were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 475.

Subcommittee B-2 on Nomenclature and Definitions (A. G. Scroggie, chairman):

This subcommittee recommends for publication as tentative several new proposed Definitions of Terms Relating to Textiles, as appended hereto.[3]

In connection with the new definitions, the subcommittee recommends that the definition of resiliency, published as a tentative revision of the Standard Definitions of Terms Relating to Textile Materials (D 123 – 42) be withdrawn as it is replaced by the new definition for resilience appearing in the new proposed definitions.

Subcommittee B-4 on Bleaching, Dyeing, and Finishing (W. M. Scott, chairman; K. H. Barnard, acting chairman):

This subcommittee recommends that the following tentative standards be continued as tentative for the reasons given:

Tentative Method of Test for Volumetric Determination of Small Amounts of Copper in Textiles (D 463 – 37 T), pending the results of a further study to determine the advisability of retaining it,

Tentative Method of Test for Apparent Fluidity of Dispersions of Cellulose Fibers in Cuprammonium Hydroxide (D 539 – 40 T), pending reconciliation of differences between laboratories using this method,

Tentative Methods of Test for Resistance of Textile Fabrics to Water (D 583 – 40 T), pending results of work in progress on proposed revisions,

Tentative Method of Test for Resistance of Textile Fabrics and Yarns to Insect Pests (D 582 – 41 T), pending results of interlaboratory tests on a variety of fabrics and several types of moth-repellents,

Tentative Specifications for Fire-Retardant Properties of Treated Textile Fabrics (D 626 – 41 T), pending study of a variety of new materials coming into use for military purposes, and

Tentative Methods of Test for Evaluating Compounds Designed to Increase Resistance of Fabrics and Yarns to Insect Pests (D 627 – 41 T), pending the more complete standardization of methods of testing resistance to insects in Tentative Methods D 582 – 41 T.

The subcommittee has formulated plans for sponsoring a very important and urgently needed research on accelerated aging tests.

Subcommittee C-2 on Papers (R. H. Brown, chairman):

This subcommittee arranged for the presentation of the following papers at the March, 1943, meeting:[4]

"Progress in New Synthetic Textile Fibers,"[5] by H. R. Mauersberger,
"Standards Division Operations, Office of Price Administration," by H. S. Schenker, and
"The Balls Sledge Cotton Sorter," by R. W. Vose.

The following several new fibers and products made from them were exhibited through the courtesy of the companies indicated:

"Soylon" (soybean fiber), Ford Motor Co.
"Aralac" (Casein fiber), National Dairy Products Corp.
"Velon" (vinylidene chloride resin), Firestone Tire and Rubber Co. and Hafner Associates.
"Vinyon" (Vinylite resin yarns of elastic and nonelastic qualities), American Viscose Corp.
Plastic- or resin-coated rayon and cotton yarns, Freydberg Bros.-Strauss, Inc.

This report has been submitted to letter ballot of the committee which consists of 260 members; 96 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. J. BALL,
Chairman.

W. H. WHITCOMB,
Secretary.

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[3] These definitions were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 496.

[4] Abstracts of these papers appear in the special compilation "A.S.T.M. Standards on Textile Materials," October, 1943, pp. 407, 412, and 415, respectively.

[5] Published in ASTM BULLETIN, No. 122, May, 1943, p. 25.

APPENDIX

PROPOSED REVISIONS IN STANDARDS FOR TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentative standards covering textile materials which are referred to earlier in this report.[1] These standards and tentative standards appear in their present form in the 1942 Book of A.S.T.M. Standards, Part III.

REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Methods of Testing and Tolerances for Yarns Spun from Wool Mixed with Fibers Other than Wool (D 508 - 39):

Title.—Change to read as follows by the omission of the words in brackets:

> Standard Methods of Testing and Tolerances for Yarns Spun from [Wool] Mixed [with] Fibers [Other than Wool]

Section 1 (a).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> 1. These methods of testing and tolerances apply to single or plied yarns of any of the following types spun from [wool mixed with fibers other than wool] *mixed fibers of different types:*
>
> (*a*) Yarns Spun on the Woolen System:
>
> Scoured Spun-in-Oil Yarn, made from thoroughly scoured [wool] *fibers* to which oil has been added,
>
> Semiscoured Spun-in-Oil Yarn, [made from] *only applicable when* wool [which] has been washed at the source but not scoured, and to which oil has been added, and
>
> Dry-Spun Yarn, made from thoroughly scoured [wool] *fibers* without the addition of oil.

Section 6.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*a*) Yarn Number.—The number of a yarn spun from [wool] mixed *fibers* [with fibers other than wool] *of different types* shall be the number of thousands of yards per pound (typp system).

Change the first sentence of Paragraph (*c*) to read as follows by the omission of the words in brackets:

> Standard condition of a yarn spun from [wool] mixed [with] fibers [other than wool] shall be that reached by the yarn when in moisture equilibrium with a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.).

In Paragraph (*e*) add to the tabulation of fibers a new fiber "aralac" with a commercial regain of 13 per cent.

Change the first sentence of Paragraph (*f*) to read as follows by the omission of the words in brackets:

> The commercial regain of a yarn spun from [wool] mixed [with] fibers [other than wool] shall be that calculated from the commercial regains of the fibers used in proportion to the percentage composition based on the dry fiber content by means of the following formula.

In the Note following Paragraph (*f*) change the parenthetical phrase "dry spun, French system" to read "dry basis."

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[1] See p. 385.

Section 7.—Change the first sentence to read as follows by the omission of the words in brackets:

> Yarn spun from [wool] mixed [with] fibers [other than wool] shall conform to the tolerances prescribed in Sections 8 to 11.

Section 20 (*a*) and (*b*).—In the last sentence of each of these paragraphs, change the drying temperature from "100 to 105 C." to read "105 to 110 C."

TENTATIVE REVISIONS OF STANDARDS

Standard Specifications and Methods of Test for Asbestos Tape for Electrical Purposes (D 315 - 41):

Section 18 (*a*).—Change from its present form: namely,

> (*a*) The thickness shall be measured in accordance with Method B of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials.

to read as follows:

> (*a*) The thickness shall be measured in accordance with Section 5 of the Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39) of the American Society for Testing Materials.

Standard Methods of Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 - 42):

Section 15 (*a*).—Change from its present form to read as follows:

> 15. (*a*) *Testing Machine.*—A single strand machine of one of the following types shall be used: Type A, pendulum type; or type B, constant specimen-rate-of-load type. The machine shall conform to the requirements prescribed in the Standard Specifications for Textile Testing Machines (A.S.T.M. Designation: D 76) of the American Society for Testing Materials. It shall be equipped with an autographic recorder and with clamps of the cam type having spools not less than ½ in. in diameter. The clamps shall be so arranged to permit the application of an initial tension of 4 oz. to the free end of the cord before closing the second clamp. There shall be 10 in. between the nips of the clamps measured along the line of the cord. The capacity of the machine shall be as follows:

Breaking Strength, lb.	Machine Capacity, lb.
20 and under..........	25
Over 20...............	50

> NOTE.—Due to the different principles of machine design involved, it is not possible to correlate test results obtained on type A machines with those obtained on type B machines. Comparison of test results should be confined to tests made on the same type of machine.

Standard Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 - 42):

Section 19 (*a*).—Change the second sentence to read as follows by the addition of the italicized words:

> The distance between the clamps shall be 18 in. *when a pendulum type tester is used, and 20 in. when a constant specimen-rate-of-load tester is used.*

Standard Methods of Testing and Tolerances for Spun Rayon Yarns and Threads (D 507 - 42):

New Section.—Add a new Section 23 on grades to read as follows:

> 23. *Grades.*—(*a*) Photographic standards have been established for cotton yarns[2] in grades A, B, C, and D, where A is the highest grade and the others are progressively lower. It is recognized that many spun rayon yarns are of a better grade than grade A, therefore, an additional grade AA has been established for yarns grading better than A. As soon as conditions permit, photographic standards will be prepared for grade AA. Although only five grades are thus provided, it is feasible to recognize thirds of grades, such as, AA plus, AA, AA minus; A plus, A, A minus; B plus, B, B minus, etc., so that fifteen distinct gradations may be employed in grading yarns. These appearance standards may be used for either carded or combed single yarns. Yarns shall be graded separately for eveness and for neps, since it is possible for a yarn to grade differently for each of these two characteristics. These grades apply to several groups of yarn numbers for each of

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[2] See Standard General Methods of Testing and Tolerances for Cotton Yarns (A.S.T.M. Designation: D 180) of the American Society for Testing Materials, 1942 Book of A.S.T.M. Standards, Part III, p. 607.

which there is a separate set of standards. The grouping of yarn numbers is as follows:

3.0's to 7.0's
7.0's to 16.5's
16.5's to 32.0's
32.0's to 65.0's
65.0's to 125.0's

(*b*) *Procedure.*—A sample of the yarn under test shall be wound upon a black board of designated size with a designated traverse and compared with a photographic standard (Note).

(*c*) *Number of Tests.*—Five tests shall be made on each case, bale, chain ball, or beam warp. If 80 per cent of the results meet the specified grade and the remaining 20 per cent do not fall below the next lower grade, the yarn shall be considered to be of the grade specified.

NOTE.—A set of photographic standards for each group of yarns, together with detailed information regarding the size of board and traverse, may be obtained at the nominal cost of $10.00 from the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

Tentative Methods of Testing and Tolerances for Woven Glass Fabrics (D 579 - 41 T):

Section 10 (*a*).—Change to read as follows by the addition of the italicized word and letters:

(*a*) The thickness of the fabric shall be determined in accordance with method A, B, or *C* of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials. In case of dispute method A *or C* shall be used.

Tentative Methods of Testing and Tolerances for Woven Glass Tapes (D 580 - 41 T):

Section 11 (*a*).—Make the same change in this section as is recommended above in Section 10 (*a*) of Methods D 579 - 41 T.

EDITORIAL CHANGE IN TENTATIVE STANDARD

Tentative Methods of Testing and Tolerances for Glass Yarn (D 578 - 40 T):

Section 2 (*e*).—Omit the definition for equivalent single number and the note following the paragraph, since this definition is not applicable to glass yarn.

REPORT OF COMMITTEE D-18

ON

SOILS FOR ENGINEERING PURPOSES

Committee D-18 on Soils for Engineering Purposes has held two meetings during the past year: in Atlantic City, N. J., on June 23, 1942, and in St. Louis, Mo., on December 4, 1942.

SYMPOSIUM ON TESTS FOR SOILS

A preliminary session on the Symposium on Tests of Soils for Engineering Purposes on which the committee has been working for the past two years will be presented at this annual meeting of the Society being held in Pittsburgh, Pa., from June 28 to July 2, 1943. In preparation for this symposium a number of individuals have submitted descriptions of various test procedures for soils with which they are familiar. Following a presentation by Mr. Paul Rapp acting for the chairman of the Symposium Committee, it is proposed to call upon a number of individuals to introduce the several groups of tests: Indication Tests —C. A. Hogentogler; Compaction Test —K. B. Woods; Shear Test—E. J. Kilcawley; Test for Bituminous Mixtures—F. V. Reagel; and Test for Soil Cement Mixtures—M. D. Catton.

The descriptions of test procedures which have been prepared for the Symposium have been circulated in the committee and comments on them received from members. They have, however, not been preprinted.[1]

RECOMMENDATIONS AFFECTING STANDARDS

The committee has no recommendations to make at this time with respect to the standards and tentative standards under its jurisdiction.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee activity during the year has in the main been confined to the work done by individual members on tests to be described in the Symposium.

Subcommittee VIII on Methods of Testing for Mechanical Stability of Soils and Subcommittee IX on Methods of Testing for Bearing Capacity of Soils in Place (Load Tests) have until recently had no chairmen, as the former chairmen L. W. Hamilton and W. S. Housel are serving in the Armed Forces. R. F. Blanks and T. A. Middlebrooks have since been appointed to serve as chairmen, respectively, of these two subcommittees. Committee D-18 has also lost for the duration of the war, a number of other members.

This report has been submitted to letter ballot of the committee which consists of 66 voting members; 52 members returned their ballots, of whom 47 have voted affirmatively and 5 negatively.

Respectfully submitted on behalf of the committee,

C. A. HOGENTOGLER,
Chairman.

WESLEY R. NELSON,
Secretary.

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[1] See Note on the Symposium on Tests of Soils for Engineering Purposes, p. 394.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-18 presented to the Society through Committee E-10 on Standards a proposed tentative revision of the Standard Method of Mechanical Analysis of Soils (D 422 – 39). This tentative revision was accepted by Committee E-10 on August 30, 1943, and appears in the 1943 Supplement to Book of A.S.T.M. Standards, Part II, p. 163.

NOTE ON THE SYMPOSIUM ON TESTS OF SOILS FOR ENGINEERING PURPOSES

As indicated in the report of Committee D-18, a number of descriptions of test procedures had been submitted by various individuals. It is proposed to issue these, together with tests that have already been standardized by the Society, as a complete compilation of soil test procedures, looking toward the holding of a formal discussion on these procedures at a forthcoming annual meeting of the Society. A list of these procedures is given below. It is expected that the published compilation will appear during the coming year.

Indicator Tests on Soils:

Tentative Methods of Surveying and Sampling Soils for Highway Subgrades (D 420 – 42 T).
Standard Method of Preparing Soil Samples for Mechanical Analysis and Determination of Subgrade Soil Constants (D 421 – 39).
Suggested Wash Method of Sample Preparation for Stabilization Materials and Stabilization Mixes.
Standard Method of Mechanical Analysis of Soils (D 422 – 39) and Suggested Modifications.
Suggested Method for Mechanical Analysis of Soil by Elutriation.
Standard Method of Test for Liquid Limit of Soils (D 423 – 39).
Standard Method of Test for Plastic Limit and Plasticity Index of Soils (D 424 – 39).
Suggested Alternate Method of Test for Plastic Limit and Plasticity Index of Soils.
Standard Method of Test for Centrifuge Moisture Equivalent of Soils (D 425 – 39).
Standard Method of Test for Field Moisture Equivalent of Soils (D 426 – 39).
Standard Method of Test for Shrinkage Factors of Soils (D 427 – 39).
Suggested Method of Test for Permeability of Porous Granular Material.
Suggested Test for Capillary Rise.

Compaction Tests on Soils:

Suggested Method of Test for Consolidation of Cohesive Soils.
Suggested Method of Test for Consolidation of Soil.
Suggested Methods of Test for Soil Compaction.
Tentative Method of Test for Moisture-Density Relations of Soils (D 698 – 42 T).
Suggested Method of Test for Determination of Volume Change of Soils.

Strength Tests on Soils:

Suggested Method of Test for Direct Shear in Cohesive Soils by the Box Shear Method.
Suggested Method of Test for Direct Shear in Noncohesive Soils by the Box Shear Method.
Suggested Method for the Determination of the True Shearing Resistance of Soil Samples by the Direct Shear Method.
Suggested Methods of Tests for Soils and Soil Mixtures.
Suggested Method of Direct Shear Test of Soil.
Suggested Method for Triaxial Compression Tests.
Suggested Method of Test for Bearing Ratio and Expansion of Soils.
Suggested Method for Determining Bearing Power of Soil by Means of a Cone.
Suggested Method for Determination of the Shear Strength of Flexible Road Surfaces, Subgrades, and Fills by the Burggraph Shear Test.
Suggested Method for Saturating Strength Test Specimens of Cohesive Soil.

Soil-Cement Tests:

Suggested Method of Test for Determining Cement Content of Soil-Cement Mixtures.
Tentative Method of Test for Moisture-Density Relations of Soil-Cement Mixtures (D 558 – 40 T).
Tentative Method of Wetting-and-Drying Test of Compacted Soil-Cement Mixtures (D 559 – 40 T).
Tentative Method of Freezing-and-Thawing Test of Compacted Soil-Cement Mixtures (D 560 – 40 T).

Bituminous Mixtures:

Suggested Field Procedure for the Design of Cut-Back Asphalt-Soil Mixtures.

Suggested Method of Test for the Determination of Suitable Proportions of Soil-Asphalt or Soil-Aggregate Asphalt Mixtures.

Suggested Method of Test for Stability of Mixtures of Soils and Liquid Asphaltic Products.

Suggested Method of Making Bearing Value Tests on Soils for Use in Sand-Bituminous Road Mix.

Suggested Procedure for Arriving at Recommendations for Construction of Tar Stabilized Soil Bases, and Suggested Methods of Test for Evaluating Tar Soil-Mixtures.

Suggested Method of Test for Determining the Modified Bearing Value of Soil-Bituminous Mixtures.

Suggested Method of Test for the Determination of the Quantity of Cut-Back Asphalt for Stabilizing Sandy Soils.

Suggested Method of Test for Water Absorption and Resistance to Plastic Flow of Soil and Emulsified Asphalt Mixtures.

Suggested Bearing Value Test for Sand-Asphaltic Mixtures.

Suggested Method of Test for Asphalt Soil Stabilization Design.

Suggested Method for Testing Soil-Bituminous Mixtures by Means of the HVEEM Stabilometer.

Suggested Method of Test for Soil-Bitumen Mixtures in the Free Rubber Closed System Stabilometer.

Suggested Combined Test for Determining the Volume Change and Loss of Stability in Bituminous-Soil Mixtures in the Presence of Water.

Suggested Method of Test for Stabilization of Soils with Bituminous Materials.

Suggested Method of Test for Water Absorption, Volume Change, and Stability of Mixtures of Soil and Tar.

REPORT OF COMMITTEE D-19

ON

WATER FOR INDUSTRIAL USES

Committee D-19 on Water for Industrial Uses and its subcommittees held meetings at Atlantic City, N. J., on June 22 and 23, 1942. No additional meetings were held by any of the subcommittees during the year.

The committee sponsored a session on water at the 1942 annual meeting in the form of a Round-Table Discussion on The Solvent Action of Water Vapor at High Temperatures and Pressures. The principal paper[1] introducing the discussion was presented by G. W. Morey, Geophysical Laboratory, Carnegie Institution of Washington, Washington, D. C.

The committee is sponsoring a water session at this annual meeting of the Society, the program comprising two papers on determination of dissolved oxygen, to be followed by a Symposium on the Identification of Water-Formed Deposits, Scales and Corrosion Products by Physico-Chemical Methods. The papers[2] comprising the session are as follows:

"Field and Laboratory Determination of Dissolved Oxygen," by R. C. Adams, U. S. Naval Engineering Experiment Station, Robert E. Barnett, U. S. Army, and D. E. Keller, Jr., U. S. Naval Engineering Experiment Station.

"Applicability of the Schwartz-Gurney Method for Determining Dissolved Oxygen in Boiler Feedwater and Modification of the Method to Make it Especially Applicable in the Presence of Such Impurities as Are Encountered in Power Plants," by R. C. Ulmer, J. M. Reynar, and J. M. Decker, The Detroit Edison Co.

Symposium on the Identification of Water-Formed Deposits, Scales, and Corrosion Products by Physico-Chemical Methods

Introduction to Symposium by H. C. Miller, Public Service Electric and Gas Co.

"X-Ray Diffraction Methods in the Study of Power Plant Deposits," C. E. Imhoff and L. A. Burkhardt, Allis-Chalmers Mfg. Co.

"Diagnosis of Water Problems at Limbo Station," E. P. Partridge, R. K. Scott, and P. H. Morrison, Hall Laboratories, Inc.

"The Interpretation of Analyses and Problems Encountered in Water Deposits," J. A. Holmes and A. O. Walker, National Aluminate Corp.

The introduction by H. C. Miller will include the results secured by five laboratories on two samples of deposits, illustrating the increased information that is secured when modern laboratory tools are utilized in the analysis and identification of substances. In addition to macro-chemical analyses reported, there will be found the results of microanalysis as well as X-ray analysis.

The Society was represented by Max Hecht at a meeting of the Subcommittee on Care of Steam Boilers in Service of the Boiler Code Committee, of The American Society of Mechanical Engineers, held in New York, N. Y., on December 2, 1942. The following assignments are being worked on actively affecting the paragraphs on water chemistry that now appear in the A.S.M.E. Boiler Construction Code, 1940 Edition, and especially Section VII on Suggested Rules for Care of Power Boilers: embrittlement and cracking, cleaning out and laying up of boilers, scale sludge and oil, priming and carry-over, corrosion, sampling, testing, and reporting.

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[1] George W. Morey, "Solubility of Solids in Water Vapor," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 980 (1942).

[2] See pp. 1240 to 1310.

Suggestions from members of the Society and others having experience in boiler operation are solicited and should be addressed to the Society representative.

The Society was represented by Max Hecht and E. P. Partridge, the latter representing F. N. Speller, at the meeting of the Executive Subcommittee of the Joint Research Committee on Boiler Feedwater Studies, held on December 4, 1942, in New York, N. Y.

Committee D-19 was represented by H. C. Miller at the annual meeting of the Water Committee of the Technical Association of Pulp and Paper Industries, held in New York, N. Y., on February 15, 1943. Mr. Miller outlined the scope and activities of Committee D-19 to this group.

A general statement on reporting the results of water analysis was published in the October, 1942, issue of *Mechanical Engineering,* under the title "Ions, Parts Per Million and Equivalents Per Million."

The committee has made important changes in the scope of the standing committee and of several of its subcommittees, has organized a new Subcommittee on Methods of Testing, and has reorganized several of its subcommittees. The changes which have received unanimous approval by letter ballot vote of the committee show the wider field of activity that the committee finds it necessary to enter. The scope of the main committee has been changed to read as follows by the addition of the italicized words:

> The study *and description* of water as an engineering material for industrial purposes. *This will include* terminology; methods of sampling, analysis, *and testing*; interpretation of results of *analyses* and tests; classification; and *the preparation of* specifications.

I. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following two tentative standards be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Method for Determination of Total Carbon Dioxide and Calculation of the Carbonate and Bicarbonate Ions in Industrial Waters (**D 513 – 38 T**):[3]

Fig. 2.—Add the following after the figure caption:

$a = CO_3^{--}$ $b = HCO_3^-$ $c = H_2CO_3$

Footnote 2.—Replace the present reference by the following:

> [2] D. S. McKinney, "Calculation of Corrections to Conductivity Measurements for Dissolved Gases," *Proceedings,* Am. Soc. Testing Mats., Vol. 41, p. 1285 (1941).

Section 6 (*b*).—Add to this section an additional note reading as follows:

> NOTE.—For greater accuracy the curves in Fig. 2 may be reproduced on a larger scale from the data given in the following table:

pH	Fraction of Total Carbon Dioxide[a] Present As		
	H_2CO_3	HCO_3^-	CO_3^{--}
2.0......	1.0000	...	...
2.5......	0.9999	0.0001	...
3.0......	0.9996	0.0004	...
3.5......	0.9986	0.0014	...
4.0......	0.9957	0.0043	...
4.5......	0.9866	0.0134	...
5.0......	0.9587	0.0413	...
5.5......	0.8800	0.1200	...
6.0......	0.6988	0.3012	0.0000
6.5......	0.4232	0.5767	0.0001
7.0......	0.1883	0.8113	0.0004
7.5......	0.0683	0.9303	0.0014
8.0......	0.0226	0.9728	0.0046
8.5......	0.0072	0.9783	0.0145
9.0......	0.0022	0.9530	0.0448
9.5......	0.0006	0.8701	0.1293
10.0......	0.0002	0.6801	0.3197
10.5......	0.0000	0.4022	0.5978
11.0......	...	0.1754	0.8246
11.5......	...	0.0630	0.9370
12.0......	...	0.0208	0.9792
12.5......	...	0.0067	0.9933
13.0......	...	0.0021	0.9979

[a] These values have been calculated from dissociation constants given in W. M. Latimer's "Oxidation Potentials," Prentice Hall, Inc., 1938, New York, N. Y.

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[3] 1942 Book of A.S.T.M. Standards, Part III, p. 1549.

Tentative Method for Determination of the Total Orthophosphate and Calculation of the Respective Orthophosphate Ions in Industrial Waters (**D 515 – 40 T**):[4]

Fig. 1.—Replace this figure by the accompanying Fig. 1:

Footnote 3.—Replace the present reference by the following:

> [3] D. S. McKinney, "Calculation of Corrections to Conductivity Measurements for Dissolved Gases," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 1285 (1941).

Section 5 (b).—Add to this section an additional note reading as follows:

> NOTE.—For greater accuracy the curves of Fig. 1 may be reproduced on a larger scale from the data given in the following table:

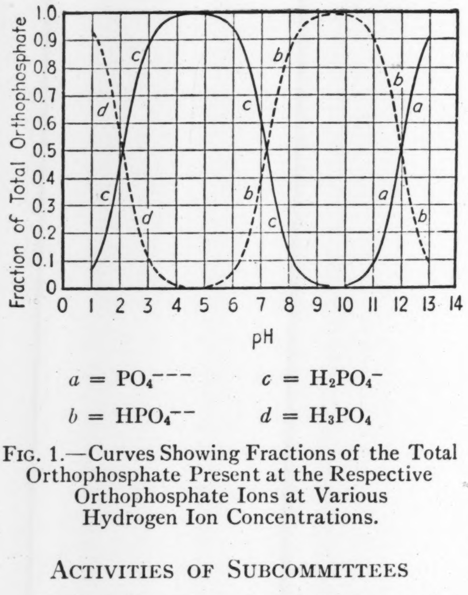
pH	Fraction of Total Orthophosphate[a] Present As			
	H_3PO_4	$H_2PO_4^-$	HPO_4^{--}	PO_4^{---}
1.0	0.9302	0.0698	...	...
1.5	0.8083	0.1917	...	...
2.0	0.5714	0.4286	...	...
2.5	0.2966	0.7034	0.0000	...
3.0	0.1176	0.8823	0.0001	...
3.5	0.0405	0.9593	0.0002	...
4.0	0.0132	0.9862	0.0006	...
4.5	0.0042	0.9939	0.0019	...
5.0	0.0013	0.9925	0.0062	...
5.5	0.0004	0.9804	0.0192	...
6.0	0.0001	0.9415	0.0584	...
6.5	0.0000	0.8361	0.1639	...
7.0	...	0.6173	0.3827	...
7.5	...	0.3378	0.6622	0.0000
8.0	...	0.1390	0.8609	0.0001
8.5	...	0.0485	0.9512	0.0003
9.0	...	0.0159	0.9831	0.0010
9.5	...	0.0051	0.9918	0.0031
10.0	...	0.0016	0.9885	0.0099
10.5	...	0.0005	0.9689	0.0306
11.0	...	0.0001	0.9090	0.0909
11.5	...	0.0000	0.7598	0.2402
12.0	...	...	0.5000	0.5000
12.5	...	...	0.2403	0.7597
13.0	...	...	0.0909	0.9091

[a] These values have been calculated from dissociation constants given in W. M. Latimer's "Oxidation Potentials," Prentice Hall, Inc., 1938, New York, N. Y.

An editorial correction was included in Method D 515 before publication in the 1942 Book of A.S.T.M. Standards. In Section 3 (*c*) describing the magnesia mixture the second sentence was changed to read as follows by the addition of the italicized figures and the omission of those in brackets:

> Add to this solution [250] *140* g. of NH_4Cl, reagent grade, and [700] *350* ml. of NH_4OH (sp. gr. 0.90), reagent grade, and dilute to 1 liter.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 70 members; 61 members returned their ballots, of whom 51 have voted affirmatively, 0 negatively, and 10 members marked their ballots "not voting" with respect to Tentative Method D 513, and 46 have voted affirmatively, 0 negatively, and 15 members marked their ballots "not voting" with respect to Tentative Method D 515.



$a = PO_4^{---}$ $c = H_2PO_4^-$

$b = HPO_4^{--}$ $d = H_3PO_4$

FIG. 1.—Curves Showing Fractions of the Total Orthophosphate Present at the Respective Orthophosphate Ions at Various Hydrogen Ion Concentrations.

ACTIVITIES OF SUBCOMMITTEES

Advisory Committee (Max Hecht, chairman).—Two meetings were held during the year for transaction of business, one in June, 1942, at Atlantic City, N. J., the other in November, 1942, at Pittsburgh, Pa.

Subcommittee II, Editorial (E. P. Partridge, chairman).—This subcommittee held a meeting in June, 1942, at Atlantic City, N. J., at which the chairman outlined the procedure followed by the subcommittee in developing and reviewing material for publication.

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[4] 1942 Book of A.S.T.M. Standards, Part III, p. 1552.

Subcommittee III on Methods of Sampling (D. K. French, chairman).—The subcommittee held one meeting during the year, in June, 1942, at Atlantic City, N. J. During the year it made a further study of suggestions submitted by members on the sampling of boiler feedwater, boiler water, and steam. These suggestions will be presented to the members of the subcommittee during the current meeting for development, if possible, into tentative methods for both control and referee procedures.

Subcommittee IV on Methods of Analysis (H. C. Miller, chairman).—The subcommittee held one meeting during the year, in June, 1942, at Atlantic City, N. J. During the year, reorganization of the subcommittee was effected to permit greater ease and speed of handling the large number of methods of analysis for which this subcommittee is responsible. While emphasis has in the past been placed on gravimetric and macro-chemical procedures, rapid development of new analytical techniques indicated the desirability of a new functional set-up of the subcommittee to permit it to better serve the Society and industry.

Laboratory methods of analysis have been divided into three main sections: analytical, electrometric, and special, each under a section chairman, and an important new section on field tests has been added. As many of the members of the committee have specialized in various analytical techniques, it is believed that such talent will be most effectively used in the new grouping. The program of work for these new sections will be developed at the time of this annual meeting.

Subcommittee V on Classification (W. C. Schroeder, chairman).—This subcommittee held one meeting, in June, 1942, at Atlantic City, N. J. During the year it has been further exploring the manner of classification, and at the Pittsburgh meeting the results of this year of study will be reviewed.

Subcommittee VI on Methods of Testing (V. V. Kendall, chairman).—This subcommittee was formed during the year, under the direction of Mr. Kendall, Corrosion Engineer, National Tube Co. It has undertaken the formulation of standard methods of measuring the behavior of water with particular respect to the corrosivity and stability under various conditions of use, where such standardization can be made. It is also undertaking a survey of the various methods of testing the corrosivity of water in its various states, with a view to the formulation of a manual or guide consisting of a description of each test, its application to given conditions, and the results to be obtained by its use.

This report has been submitted to letter ballot of the committee which consists of 65 members, 55 returned their ballots, of whom 51 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

R. E. HALL,
Secretary.

APPENDIX

SCOPE OF COMMITTEE D-19 AND ITS SUBCOMMITTEES

REVISED SEPTEMBER 1, 1942

Committee D-19 on Water for Industrial Uses has as its purpose the study and description of water as an engineering material for industrial purposes. This will include terminology; methods of sampling, analysis, and testing; interpretation of results of analyses and tests; classification; and the preparation of specifications.

Subcommittee I, Advisory:

The purpose of this subcommittee is to act on recommendations and matters referred to the officers from the Society as well as from Committee D-19.

Subcommittee II, Editorial:

The function of this subcommittee is the preparation in standard form of specifications and methods of sampling, analysis and testing which are approved by the standing committee, including interpretation of the results of individual test methods and the terminology employed in the description of the methods.

Subcommittee III on Methods of Sampling:

The scope of this subcommittee includes the preparation of standard methods for the sampling of water in its various states, and of the deposits resulting from or incident to the industrial use of water. Within its province is the sampling of:

1. Surface and subsurface water supplies and water in tanks.
2. Water and water vapor flowing in pipes.
3. Water and water vapor subjected to atmospheric, subatmospheric and superatmospheric pressures and/or atmospheric, subatmospheric and superatmospheric temperatures. Methods to be submitted shall include those for natural and processed waters for industrial uses, water vapor and condensation products of water vapor.

Also within its province is the sampling of scales, corrosion products or organic growths formed during the industrial use of water.

Subcommittee IV on Methods of Analysis:

The scope of this subcommittee includes the collection and verification of data for use in the preparation of standard methods of analysis of water in its various states and of the deposits resulting from or incident to the industrial use of water. It shall furnish methods for the determination of the various ions, dissolved gases and other substances in water having both a varied H-ion concentration range and a varied dissolved salt content. These methods should be distinguished for use in concentrated products, natural supply, and processed supply. Both referee and field methods shall be formulated.

Subcommittee IV shall also furnish methods for the identification by special means such as the microscope, X-ray, or spectrograph, of the constituents of deposits from water.

Subcommittee V on Classification:

One function of this subcommittee is the development of standard specifications for expressing analytical data. A second function is the development of standard methods for classifying the probable behavior of water with respect to corrosion, deposition of solids when heated or processed, or other effects which are significant in determining the suitability of the water for industrial use.

Subcommittee VI on Testing:

The scope of this subcommittee comprises the formulation of standard methods of measuring the behavior of water with particular respect to corrosivity and stability under various conditions of use. Methods of testing to be sponsored by this subcommittee shall be considered to be procedures which will evaluate the combined effects of water under a specific set of conditions and not necessarily the evaluation of the effect of a specific constituent of the water.

The subcommittee shall survey the various methods of testing and the data obtained by these methods for the corrosivity of water in its various states, with a view to the formulation of a manual or guide consisting of the description of each test, its application to given conditions, and the results to be observed by its use. The subcommittee shall likewise survey methods of testing the tendency of water to form deposits under conditions of industrial use, including sedimentation and flocculation as well as the formation of scales and organic growths.

REPORT OF COMMITTEE D-20

ON

PLASTICS

Since the June, 1942, meeting in Atlantic City, N. J., Committee D-20 on Plastics and its subcommittees have held two meetings: in New York, N. Y., in October, 1942, and March, 1943.

Three new subcommittees have been organized: Subcommittee VII on Analytical Methods, P. D. Brossman, chairman; Subcommittee VIII on Research under the chairmanship of H. K. Nason; and Subcommittee IX on Molds and Molding Processes under the temporary chairmanship of Robert Burns.

W. H. Gardner has resigned as chairman of Subcommittee II on Hardness, and L. Boor has been appointed in his place.

Committee D-20 now has 94 voting members, representing an increase during the year of 13 voting members.

The committee has sponsored a special compilation of "A.S.T.M. Standards on Plastics" which contains all of the specifications and test methods developed by the committee and in addition includes a number of related standards prepared by several other A.S.T.M. committees that are of interest in the testing of plastics.

Subsequent to the 1942 annual meeting, Committee D-20 presented to the Society, through Committee E-10 on Standards, 10 new tentative specifications, 3 new tentative methods, and revisions in 2 tentative standards. These various recommendations are listed in Table I together with the results of the letter ballot vote in Committee D-20 and

TABLE I.—RECOMMENDATIONS ACCEPTED BY COMMITTEE E-10 ON STANDARDS, INCLUDING RECORD OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
New Tentative Specifications for:			
Phenolic Molding Compounds (D 700 – 43 T), *accepted March 24, 1943*	47	3	18
Cellulose Nitrate (Pyroxylin) Plastic Sheets, Rods and Tubes (D 701 – 43 T), *accepted March 11, 1943*	29	3	21
Cast Methacrylate Plastics (D 702 – 43 T), *accepted March 11, 1943*	29	4	20
Polystyrene Molding Compounds (D 703 – 43 T), *accepted March 11, 1943*	29	5	19
Melamine-Formaldehyde Molding Compounds (D 704 – 43 T), *accepted March 11, 1943*	28	6	19
Urea-Formaldehyde Molding Compounds (D 705 – 43 T), *accepted March 11, 1943*	30	6	17
Cellulose Acetate Molding Compounds (D 706 – 43 T), *accepted March 20, 1943*	37	5	12
Cellulose Acetate Butyrate Molding Compounds (D 707 – 43 T), *accepted March 20, 1943*	32	3	19
Rigid Sheets made from Vinyl Chloride - Acetate Resins (D 708 – 43 T), *accepted March 20, 1943*	35	1	18
Laminated Thermosetting Materials (D 709 – 43 T), *accepted April 5, 1943*	39	2	13
New Tentative Methods of Test for:			
Compressive Strength of Plastics (D 695 – 42 T), *accepted August 24, 1942*	39	0	3
Coefficient of Linear Thermal Expansion of Plastics (D 696 – 42 T), *accepted August 24, 1942*	38	0	4
Water Vapor Permeability of Plastic Sheets (D 697 – 42 T), *accepted August 24, 1942*	32	0	10
Revision of Tentative Method of:			
Test for Tensile Properties of Plastics (D 638 – 42 T), *accepted August 24, 1942*	34	1	7
Additions to Tentative:			
Descriptive Nomenclature of Objects Made from Plastics (D 675 – 43 T), *accepted March 11, 1943*	45	0	7

the date of acceptance by Committee E-10. The new and revised tentative standards have been assigned the A.S.T.M. serial designations indicated and appear in the compilation of A.S.T.M. Standards on Plastics, May, 1943.

I. NEW TENTATIVE STANDARDS

The committee recommends that the following new specifications and methods be accepted for publication as tentative, as appended hereto:[1]

II. REVISION OF TENTATIVE STANDARD

The committee recommends the following revision in the Tentative Specifications for Cellulose Acetate Molding Compounds (D 706 – 43 T):[2]

Table II.—Add the detail requirements for molded test specimens for the several properties and grades indicated in the accompanying Table II.

TABLE II.—ADDITIONAL DETAIL REQUIREMENTS FOR MOLDED TEST SPECIMENS.

	Type I, General Purpose				Type II' Heat Resistant		Type III, Impact Resistant				Type IV, Moisture Resistant			
	Grade MH	Grade M	Grade MS	Grade S	Grade VH	Grade H	Grade MS	Grade S	Grade VS	Grade ES	Grade VH	Grade H	Grade MH	Grade M
Heat distortion temperature, min., deg. Fahr.							125							115
Impact strength (Izod), min., ft-lb. per in. of notch, at 77 F							2.0							2.0
Impact strength (Izod), min., ft-lb. per in. of notch, at −40 F	0.4	0.4	0.4	0.4	0.3	0.3	0.5	0.5	0.5	0.5	0.4	0.4	0.4	0.4
Water absorption (24-hr. immersion), max., per cent, weight gain plus soluble matter loss							3.8							2.9
Water absorption (24-hr. immersion), max., per cent, soluble matter loss	0.5	0.7	0.9	1.1	0.3	0.3	0.8	1.0	1.2	1.5	0.2	0.3	0.5	1.0
Weight loss on heating (72 hr. at 180 F.), max., per cent	3.0	4.5	5.5	7.5	1.0	1.5	5.6	6.6	8.5	10.5	1.0	1.5	2.2	4.0
Dielectric strength (short-time test), min., v. per mil							250							

Tentative Specifications for:

Vinyl Chloride – Acetate Molding Compounds,
Vinylidene Chloride Molding Compounds.

Tentative Methods of Test for:

Shear Strength of Plastics, and
Measuring Relative Mobility of Thermosetting Molding Powder.

The proposed tentative specifications have been developed largely at the instigation of the Army, Navy, and Governmental Departments and are urgently needed. The proposed tentative methods of test are needed to supply new data in the case of shear strength and to standardize and thus simplify the testing procedure in the case of relative mobility of thermosetting molding powder.

III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following six tentative standards be approved without change for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Test for:[3]

Resistance of Plastics to Chemical Reagents (D 543 – 41 T),
Flammability of Plastics 0.050 in. and Under in Thickness (D 568 – 41 T),
Measuring Flow Temperatures of Thermoplastic Molding Materials (D 569 – 41 T),
Deformation of Plastics Under Load at Elevated Temperatures (D 621 – 41 T),
Diffusion of Light by Plastics (D 636 – 41 T), and
Surface Irregularities of Flat Transparent Plastic Sheet (D 637 – 41 T).

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[1] These specifications and methods of test were accepted as tentative by the Society and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, pp. 325, 328, 357, and 354, respectively.

[2] See compilation of "A.S.T.M. Standards on Plastics," May, 1943, p. 23; available as a separate publication.

[3] 1943 Book of A.S.T.M. Standards, Part III.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 94 voting members, with the results shown in Table III.

TABLE III.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Vinyl Chloride - Acetate Molding Compounds	30	2	29
Spec. for Vinylidene Chloride Molding Compounds[a]	29	5	28
Test for Shear Strength of Plastics	40	4	18
Measuring Relative Mobility of Thermosetting Molding Powder	33	1	28
II. REVISION OF TENTATIVE STANDARD			
Spec. for Celullose Acetate Molding Compounds (D 706 – 43 T)	38	2	23
III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Test for Resistance of Plastics to Chemical Reagents (D 543 – 41 T)	45	4	13
Test for Flammability of Plastics 0.050 in. and Under in Thickness (D 568 – 41 T)	43	2	17
Measuring Flow Temperatures of Thermoplastic Molding Materials (D 569 – 41 T)	40	2	20
Test for Deformation of Plastics Under Load at Elevated Temperatures (D 621 – 41 T)[b]	41	5	16
Test for Diffusion of Light by Plastics (D 636 – 41 T)[c]	30	4	28
Test for Surface Irregularities of Flat Transparent Plastic Sheet (D 637 – 41 T)	33	2	27

[a] The classified vote on the Proposed Tentative Specifications for Vinylidene Chloride Molding Compounds was as follows: Affirmative: 8 producers, 12 consumers, 9 general interests; Negative: 1 producer, 3 consumers, 1 general interest.

[b] The classified vote on the adoption as standard of the Tentative Method of Test for Deformation of Plastics Under Load at Elevated Temperatures (D 621 – 41 T) was as follows: Affirmative: 13 producers, 18 consumers, 10 general interests; Negative, 5 producers, 0 consumers, 0 general interests.

[c] The classified vote on the adoption as standard of the Tentative Method of Test for Diffusion of Light by Plastics (D 636 – 41 T) was as follows: Affirmative: 9 producers, 15 consumers, 6 general interests; Negative: 2 producers. 0 consumers, 2 general interests.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Properties (M. H. Bigelow, chairman) has developed Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 – 41 T) (prepared jointly with Committee D-9 on Electrical Insulating Materials), Tensile Properties (D 638 – 42 T), Compressive Strength (D 695 – 42 T), and Flexural Stress (Fatigue) Test of Plastics (D 671 – 42 T).

A Method of Test for Shear Strength has been completed and is recommended for publication as tentative as mentioned earlier in this report.

Under study are tests for determining impact strength, tensile properties, and compressive strength at high and low temperatures, tests for determining the strength properties of nonrigid plastics, test for tensile properties of thin sheet material, test for flexural strength, and test for bearing strength and impact strength at high speed or under load. Standard molds are being designed for all test specimens.

Subcommittee II on Hardness Properties (L. Boor, chairman) has developed Tentative Methods of Test for Deformation Under Load (D 621 – 41 T) and Mar Resistance (D 673 – 42 T), also a Recommended Practice for Long-Time Tension Tests (D 674 – 42 T).

This subcommittee has under consideration methods of test for Rockwell hardness, scratch resistance, creep, wear resistance of bearings, and coefficient of friction.

Subcommittee III on Thermal Properties (W. A. Zinzow, chairman) has developed Tentative Methods of Test for Flammability of Plastics 0.050 in. and Under in Thickness (D 568 – 41 T), Plastics Over 0.050 in. in Thickness (D 635 – 41 T), Flow Temperatures of Thermoplastic Molding Materials (D 569 – 41 T), and Coefficient of Linear Thermal Expansion (D 696 – 42 T).

A Method of Test for Measuring Relative Mobility of Thermosetting Molding Powder has been completed and is recommended for publication as tentative, as mentioned earlier in this report.

Under consideration are methods of test for cubical expansion, rate of burning at high temperatures (glo-bar test), and a simplified procedure for determining heat distortion.

Subcommittee IV on Optical Properties (W. F. Bartoe, chairman) has developed Standard Methods of Test for Index of Refraction (D 542 – 42), Tentative Methods of Test for Diffusion of Light by Plastics (D 636 – 41 T), Surface Irregularities of Flat Transparent Sheet (D 637 – 41 T), and Haze of Transparent Plastics by Photoelectric Cell (D 672 – 42 T), and Descriptive Nomenclature of Objects Made from Plastics (D 675 – 43 T).

Under consideration is work relative to optical clarity, transmission and reflectance, and associated characteristics of plastics.

Subcommittee V on Permanence Properties (J. H. Teeple, chairman) has developed the Standard Method of Test for Water Absorption (D 570 – 42) (prepared jointly with Committee D-9 on Electrical Insulating Materials), and Tentative Methods of Test for Resistance to Chemical Reagents (D 543 – 41 T), Colorfastness of Plastics to Light (D 620 – 41 T), Water Vapor Permeability (D 697 – 42 T), and Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 41 T) (prepared jointly with Committee D-9).

A Method of Test for Resistance of Plastics or Plastic Parts to Extreme Service Conditions is being completed.

Under consideration are tests for accelerated weathering, permanent effect of heat and semipermanent effect of heat on plastics.

Subcommittee VI on Specifications (G. M. Kline, chairman) has completed ten new tentative specifications for various types of plastics, the titles and designations of which are given in Table I.

Specifications for Vinyl Chloride-Acetate Molding Compounds and Vinylidene Chloride Molding Compounds have been completed and are recommended for publication as tentative, as mentioned earlier in this report.

The subcommittee has also prepared specifications for the following molding compounds which will shortly be submitted to the Society for publication as tentative: Nonrigid Polyvinyl Chloride Acetate, Nonrigid Polyvinyl Butyral, Nonrigid Polyvinyl Chloride, and Nonrigid Ethyl Cellulose.[4] Under consideration are specifications for all other plastic materials in commercial use.

Subcommittee VII on Analytical Methods (P. D. Brossman, chairman) was organized shortly after the last annual meeting in response to a large number of requests for standard test methods for the analysis of plastics. The need was especially brought out by the work of Subcommittee VI on Specifications. The organization meeting was held at the October, 1942, meeting in New York, N. Y., and the subcommittee was officially set up at that time.

Although less than a year old, Subcommittee VII has already made considerable progress on test methods for methanol soluble content and viscosity of polystyrene and on a method of determining acetyl and butyryl content of cellulose acetate butyrate. It is actively engaged in tests on nitrocellulose, methacrylates, and chlorine containing resins. Also under consideration are methods for determination of plasticizer content and specific gravity.

Subcommittee VIII on Research (H. K. Nason, chairman).—The topic of research has been a lively subject of discussion with Committee D-20 for a number of years. In order to clarify the situation a committee was set up to study the problem some time last year. During the October, 1942, meeting this committee recommended that a subcommittee on research be established.

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[4] See Editorial Note, p. 406.

The recommendation was considered favorably and, as a result, Subcommittee VIII on Research was set up at that time. The organization was completed shortly thereafter and the subcommittee has now chosen its first project. Strength under impact, involving all phases of impact testing, will be studied, and the program is now being organized.

Subcommittee IX on Molding and Molding Processes (Robert Burns, temporary chairman).—The recent work of Committee D-20 in the preparation of procurement specifications has sharply emphasized the need of standardizing molds and molding processes, since the preparation of specimens has a pronounced effect on the resulting properties. For this reason, Subcommittee IX on Molds and Molding Processes was established. The membership consists of molders together with producers, consumers, and general interests groups. At present the organization is being set up and the work is being outlined.

This report has been submitted to letter ballot of the committee which consists of 94 voting members; 62 members returned their ballots of whom 53 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ROBERT BURNS,
Chairman.

L. W. A. MEYER,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-20 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:
Nonrigid Vinyl Chloride-Acetate Plastics (D 742 - 43 T),
Nonrigid Ethyl Cellulose Plastics (D 743 - 43 T),
Nonrigid Vinyl Chloride Plastics (D 744 - 43 T),
New Tentative Methods of Test for:
Brittleness of Nonrigid Plastics (D 746 - 43 T),
Stiffness in Flexure of Nonrigid Plastics (D 747 - 43 T),
Revision of Tentative Specifications for:
Polystyrene Molding Compounds (D 703 - 43 T),
Cellulose Acetate Molding Compounds (D 706 - 43 T), and
Cellulose Acetate Butyrate Molding Compounds (D 707 - 43 T).

These recommendations were accepted by Committee E-10 on August 30, 1943, and assigned the A.S.T.M. serial designations indicated above. The new and revised tentative standards appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, pp. 332, 337, 342, 351, 360, 292, 303, and 306.

Proposed Specifications for Nonrigid Vinyl Butyral Plastics were accepted by Committee E-10 on September 7, 1943, for publication as tentative. The new tentative specifications have been assigned the designation D 745 - 43 T and appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part III, p. 347.

On January 1, 1944, Committee E-10 accepted the following recommendations of Committee D-20:

Revision of Tentative Specifications for:
Polystyrene Molding Compounds (D 703 - 44 T),
Vinylidene Chloride Molding Compounds (D 729 - 44 T), and
Revision of the Tentative Method of Test for Compressive Strength of Plastics (D 695 - 44 T).

The revised specifications and method have been assigned the designations indicated above and have been issued in separate reprint form.

REPORT OF COMMITTEE E-1 ON METHODS OF TESTING

Committee E-1 on Methods of Testing held a meeting in Atlantic City, N. J., on June 24, 1942, during the annual meeting of the Society.

The Executive Committee of the Society appointed three representatives-at-large to serve on the Advisory Committee, namely, L. H. Fry, Edgewater Steel Co., D. E. Parsons, National Bureau of Standards, and T. Smith Taylor, Federal Telephone and Radio Corp., each for a three-year term, expiring in 1945.

Subsequent to the 1942 Annual Meeting, Committee E-1 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative:

Definitions with Procedures Relating to Conditioning and Weathering (E 41 - 42 T),

Recommended Practice for Characteristics of Standard Carbon Arc Accelerated Weathering Unit (E 42 - 42 T), and

Revision of Tentative Method of:

Test for Softening Point (Ball and Tapered Ring Apparatus) (E 28 - 39 T).

These recommendations were accepted[1] by Committee E-10 on August 24, 1942, and the new and revised tentative standards appear in the 1942 Book of A.S.T.M. Standards, Parts II and III.

On April 22, 1943, Committee E-10 accepted[1] proposed revisions in certain dimensional requirements for three A.S.T.M. kinematic viscosity test thermometers in the Tentative Specifications for A.S.T.M. Thermometers (E 1 - 42 T) recommended by Committee E-1. The revised tentative specifications have been published under the designation E 1 - 43 T.[2]

ACTIVITIES OF TECHNICAL COMMITTEES

Technical Committee I on Mechanical Testing (H. F. Moore, chairman):

The various sections of this technical committee this year have been at work mainly with war emergency problems, and their work has not, in general, resulted in proposals for changes in standards. Several of the sections presented revised standards, tentative standards, or new tentative standards at the 1942 meeting, which have been approved, and appear in the 1942 Book of A.S.T.M. Standards. As noted in last year's report, during war time it seems desirable to make no changes in the standards unless they are urgently needed.

Section on Tension Testing (R. L. Templin, chairman).—This section held a meeting during the Spring Meeting of the Society in Buffalo, N. Y. It has before it several problems which have arisen as a result of war conditions, including the consideration of tension test specimens for powdered metal.

Section on Indentation Hardness (J. R. Townsend, chairman).—The subgroup on hardness relationships of the hardened or heat-treated steels, under the chairmanship of R. L. Kenyon, held a meeting in Philadelphia, Pa., on January 21, 1943, and reviewed the extensive data compiled by the committee. At this meeting a proposed Tentative Hardness Conversion Table for Steels was completed. This conversion table is now the subject of letter ballot in this E-1 section and if approved, will be submitted to Committee E-1 for action and presentation to the Society through Committee E-10 on Standards for publication as tentative.[3] The committee that developed the conversion table includes representatives of the Society of Automotive Engineers, and the American Society for Metals, and it is expected that this hardness conversion table will also be approved by these societies.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee E-1 reported the following results of the letter ballot vote from a committee membership of 56: E 41 - 42 T, 35 ballots returned, affirmative 24, negative 0, ballots marked "not voting" 11; E 42 - 42 T, affirmative 23, negative 0, ballots marked "not voting" 12; E 28 - 42 T, affirmative 21, negative 0, ballots marked "not voting" 14; E 1 - 43 T, 42 ballots returned, affirmative 26, negative 0, ballots marked "not voting" 16.

[2] 1943 Supplement to Book of A.S.T.M. Standards, Part II, p. 148; Part III, p. 518.

In connection with the work of this section, the Symposium on the Significance of the Hardness Test of Metals in Relation to Design,[4] scheduled for the Fourth Session of the 1943 meeting of the Society, is of marked significance.

Section on Effect of Speed of Testing (P. G. McVetty, chairman).—The elimination of specific speed specifications from the Standard Methods of Tension Testing of Metallic Materials (E 8 - 42) has led to controversies between producers and consumers. Requests from ordnance and aircraft manufacturers for advice on proper testing speeds were considered by the section in collaboration with Subcommittee XIII on Methods of Physical Tests, of Committee A-1 on Steel. Since any emergency specification of testing speed requires action on the part of the A-1 subcommittees responsible for individual specifications, it was necessary to refer the problem to Committee A-1 for suitable action.

Section on Compression Testing (M. F. Sayre, chairman).—This section still has under consideration the revision of the Tentative Methods of Compression Testing of Metallic Materials (E 9 - 33 T).

Section on Impact Testing (W. W. Werring, chairman).—The Tentative Methods of Impact Testing of Metallic Materials (E 23 - 41 T) prepared by this section are being continued without revision.

No matters requiring the attention of the Section on Calibration of Testing Machines and Apparatus, the Section on Elastic Strength, or the Section on Flexure Testing have developed during the past year.

Technical Committee II on Consistency, Plasticity, and Related Properties (Eugene C. Bingham, chairman):

The tests conducted at Brooklyn Polytechnic Institute in connection with the project on Industrial Flow Tests (R. Burns, chairman), comprising the comparison of the ring-and-ball softening point test with viscosities determined by the parallel plate and ring and cylinder viscosimeter, were completed during the year. A detailed progress report on this research project will be presented by Mr. W. J. Zybert at the meeting of this technical committee to be held during the annual meeting.

At the request of Committee D-4 on Road and Paving Materials, work is being undertaken in cooperation with other interested standing committees on the use of the Saybolt Furol viscosimeter for tests of asphaltic materials at high temperatures.

Further interest has also been expressed by several standing committees in the standardization of methods for determining absolute viscosity. It is proposed to consider detailed descriptions of methods of test applicable to various materials of widely different types as regards their viscosity characteristics, and covering absolute viscosimeters most suitable for different viscosity ranges.

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[3] See Editorial Note, p. 410.

[4] See pp. 803 to 856.

As mentioned earlier in this report, the Tentative Method of Test for Softening Point (Ball and Shouldered Ring Apparatus) (E 28 – 42 T) was revised subsequent to the 1942 annual meeting to include the shouldered ring in place of the tapered ring formerly specified in the method.

The technical committee has under consideration certain comments that have been received from Committee E-8 on Nomenclature and Definitions concerning the Standard Definitions of Terms Relating to Rheological Properties of Matter (E 24 – 42).

Technical Committee III on Particle Size and Shape (L. T. Work, chairman):

The Tentative Method of Test for the Particle Size Distribution of Subsieve Size Particulate Substances (E 20 – 33 T) is being continued as tentative awaiting the completion of studies on the measurement of particle size by microscopic methods.

The Standard Specifications for Sieves for Testing Purposes (E 11 – 39) have proved to be as practical and effective in war as in peace time. It is believed that this has been the result of certain provisions which have been found sufficiently inclusive for any critical conditions that might arise in connection with the materials used and the methods of manufacture of sieves.

Technical Committee IX on Interpretation and Presentation of Data (H. F. Dodge, chairman):

During the past year the supply of the March, 1941, printing of the A.S.T.M. Manual on Presentation of Data became exhausted and arrangements were accordingly made for a 1943 reprinting. In this reprinting, the opportunity has been taken to include references to new articles on the subject of quality control, and on the basis of requests, to extend the table of control chart constants in Supplement B.

The technical committee has continued to serve actively in an advisory capacity during the year with respect to papers for publication and inquiries on subjects involving the application of statistical methods.

Technical Committee X on Conditioning and Weathering (Robert Burns, chairman):

The members of Technical Committee X are actively engaged, through their respective standing committees, in an effort to correlate the various weathering and conditioning procedures in A.S.T.M. standards with the requirements of the Armed Forces as expressed in Army, Navy, and Federal Specifications. Members of this technical committee are in frequent conference with representatives of the military services and are rendering every assistance possible.

Technical Committee XI on Designation and Interpretation of Numerical Requirements (J. A. Gann, chairman):

More urgent matters resulting from the war have delayed the considerations being given by this technical committee to a classification of certain expressions used in connection with tolerances or permissible variations and to the question as to what constitutes a reasonable deviation when the required precision does not call for or require an exact statement of necessary tolerances. Since these are of interest in the Tentative Recommended Practices for Designation of Numerical Requirements in Standards (E 29 – 40 T) this tentative standard is being continued without change.

Technical Committee XII on Laboratory Apparatus (W. H. Fulweiler, chairman):

The various sections of this technical committee have continued their cooperative work with other standing committees in reviewing and suggesting improvements in recommendations for laboratory apparatus appearing in new and revised methods of test. The committee has also rendered assistance on problems covering new or alternate types of apparatus for use as an emergency measure due to the scarcity of certain materials.

The Section on Thermometers (R. M. Wilhelm, chairman) held a meeting in Philadelphia, Pa., on February 23, 1943, and approved a revision in certain dimensional requirements of the specifications for three A.S.T.M. kinematic viscosity thermometers prescribed in the Tentative Specifications for A.S.T.M. Thermometers (E 1 - 42 T). The proposed changes were urgently needed at this time in order to facilitate the manufacture of these viscosity thermometers which are very difficult to make because of the high degree of precision made necessary by the requirements for temperature measurements in the Method of Test for Kinematic Viscosity (D 445 - 42 T) which requires temperatures to be determined within plus or minus 0.025 F. Since these changes will result in speeding-up production of these thermometers the revisions were presented to Committee E-10 on Standards and accepted on April 22, 1943.

The section has in preparation specifications for a set of six short range distillation thermometers for use in testing solvents.

Technical Committee XIII on Hydrogen Ion Determinations (E. B. Ashcraft, chairman):

This technical committee at its last meeting held in Atlantic City, N. J., on June 24, 1942, considered reports from representatives of various standing committees covering considerations being given to measurements of hydrogen ion concentrations. Consideration is being given to compiling information suitable for preparing a guide for the determination of pH.

This report has been submitted to letter ballot of the committee which consists of 56 members; 41 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. H. FULWEILER,
Chairman.

P. J. SMITH,
Ex-officio Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-1 presented to the Society through Committee E-10 on Standards proposed Tentative Hardness Conversion Tables for Steel (Relationship Between Diamond Pyramid Hardness, Rockwell Hardness, and Brinell Hardness). These tables were accepted by Committee E-10 on August 24, 1943, for publication as tentative and assigned the designation E 48 - 43 T. They appear in the 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 342.

REPORT OF COMMITTEE E-2
ON
SPECTROGRAPHIC ANALYSIS

Committee E-2 on Spectrographic Analysis held one meeting during the year in Atlantic City, N. J., on June 23, 1942.

Two new members were added to the committee during the year bringing the total membership to 76.

The committee has taken action to refer the following recommendations to Committee E-10:[1]

Proposed Tentative Methods of:
- Spectrochemical Analysis of Lead Alloys for Impurities,
- Spectrochemical Analysis of Tin Alloys for Impurities,

Proposed Revision of Tentative Methods of:
- Quantitative Spectrochemical Analysis of Zinc for Lead, Iron, and Cadmium (E 26 – 35 T), and
- Quantitative Spectrochemical Analysis of Zinc Alloy Die Castings for Minor Constituents and Impurities (E 27 – 40 T).

Subcommittee III on Quantitative Methods and Applications (M. L. Fuller, chairman) met at Atlantic City, N. J., on June 22, 1942. The subcommittee has been active during the year through its Groups 1, 2, 4, and 5, and this has resulted in the completion of the two new methods of spectrochemical analysis and the revisions of Methods E 26 and E 27.

Group 1 on copper, nickel and their alloys (P. A. Leichtle, chairman) held a meeting in June, 1942, at which the following three papers were presented:

- "Quantitative Spectroscopy Applied to Some Impurities in 70–30 Cupro-Nickel," by W. D. France,[2]
- "Study of the Applicability of the Pressed Pellet Electrode Method to the Spectrographic Analysis of Nickel Alloys," by C. J. Neuhaus,[2] and
- "Some Observations on the Quantitative Spectrographic Analysis of Copper Alloys," by P. A. Leichtle.

A continuation of the latter paper will be given at the June, 1943, meeting.

Group 2 on lead, tin, antimony, bismuth and their alloys (B. F. Scribner, chairman) held a meeting in June, 1942. The two proposed tentative methods of spectrochemical analysis of tin alloys and of lead alloys for impurities, mentioned earlier in this report, were presented to Subcommittee III at this meeting.

Group 4 on zinc, cadmium, and their alloys (M. L. Fuller, chairman) has repeated cooperative tests of a method for the analysis of cadmium, using a new sample of cadmium. The results indicate that the method is satisfactory. A method for the analysis of zinc using spark excitation was reviewed by the group.

Group 5 on ferrous metals (P. R. Irish, chairman) has cooperated with Subcommittee V on Standards and Pure Materials in establishing recommended sizes for spectrographic steel electrodes. Members of the group have cooperated in the preparation and testing of steel standards which are now being made available. A comprehensive survey by questionnaire was made of 70 laboratories in order to determine the equipment and methods in use in the ferrous industry.

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[1] See Editorial Note, p. 412.

[2] *Journal*, Optical Society of Am., Vol. 33, pp. 681, 167 (1943).

Subcommittee IV on Nomenclature and Definitions (J. J. Stumm, chairman) has recently been reorganized. This subcommittee will give consideration to standard nomenclature for the field of spectroscopy and will also serve as a committee on editorial review for Committee E-2. The subcommittee has made a survey by questionnaire of terms employed in spectroscopy which require definition.

Subcommittee V on Standards and Pure Materials (T. A. Wright, chairman) held a meeting in June, 1942, at which questions concerning the availability of standard metals and alloys and problems in their preparation were discussed. The size and shape of steel rod standards were considered and recommended sizes were approved.

This report has been submitted to letter ballot of the committee which consists of 76 members; 61 members returned their ballots, of whom 55 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. V. CHURCHILL,
Chairman.

B. F. SCRIBNER,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-2 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Methods of:

Spectrochemical Analysis of Lead Alloys for Impurities (E 49 – 43 T),

Spectrochemical Analysis of Tin Alloys for Impurities (E 51 – 43 T),

Revision of Tentative Methods of:

Quantitative Spectrochemical Analysis of Zinc for Lead, Iron, and Cadmium (E 26 – 35 T), and

Quantitative Spectrochemical Analysis of Zinc Alloy Die Castings for Minor Constituents and Impurities (E 27 – 40 T).

These recommendations were accepted by Committee E-10 on August 30, 1943, and the new and revised tentative standards appear in the 1943 Supplement to A.S.T.M. Methods of Chemical Analysis of Metals.

REPORT OF COMMITTEE E-3

ON

CHEMICAL ANALYSIS OF METALS

Committee E-3 on Chemical Analysis of Metals held one meeting during the year in Atlantic City, N. J., on June 23, 1942. Five division and nine subcommittee meetings were also held during the year.

Two new members were elected during the year.

Considerable time has been devoted by members of the committee to a detailed editorial review of all the analytical methods under the jurisdiction of the committee in connection with their preparation for publication in the new edition of A.S.T.M. Methods of Chemical Analysis of Metals.

Subsequent to the 1942 Annual Meeting, Committee E-3 presented to the Society through Committee E-10 on Standards the following recommendations which were accepted[1] on December 10, 1942:

New Tentative Methods for:

Chemical Analysis of Lead- and Tin-Base Solder Metal (E 46 – 42 T), to replace immediately the Tentative Methods of Chemical Analysis of Alloys of Lead, Tin, Antimony, and Copper (B 18 – 36 T), and
Chemical Analysis of Zinc-Base Die-Casting Alloys (E 47 – 42 T).

A new Tentative Method of Chemical Analysis for Selenium in Steel (E 30 – 43 T) was accepted[1] by Committee E-10 on March 6, 1943, as a tentative revision of the Standard Methods of Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 – 42).

The new tentative standards will appear in the new edition of A.S.T.M. Methods of Chemical Analysis of Metals.

On March 6, 1943, Committee E-10 accepted[1] the recommendation of Committee E-3 that the following A.S.T.M. tentative methods, which were out-of-date, be withdrawn:

Tentative Methods of:

Chemical Analysis of Manganese Bronze (B 27 – 36 T),
Chemical Analysis of Gun Metal (B 28 – 36 T),
Chemical Analysis of Bronze Bearing Metal (B 46 – 36 T).

RECOMMENDATION AFFECTING STANDARD

The committee recommends the proposed Recommended Practices for Apparatus and Reagents for Chemical Analysis of Metals[2] for publication as tentative.

This recommendation has been submitted to letter ballot of the committee which consists of 73 members; 54 members returned their ballots, of whom 51 have voted affirmatively, 0 negatively, and 3 members marked their ballots "not voting."

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee E-3 reported the following results of the letter ballot vote from a committee membership of 73: E 46 – 42 T, to replace B 18 – 36 T, 52 ballots returned, affirmative 47, negative 0, ballots marked "not voting" 5; E 47 – 42 T, affirmative 46, negative 0, ballots marked "not voting" 6; E 30 – 43 T, 53 ballots returned, affirmative 40, negative 0, ballots marked "not voting" 13; withdrawal B 27 – 36 T, B 28 – 36 T, and B 46 – 36 T, 58 ballots returned, affirmative 54, negative 1, ballots marked "not voting" 3.

[2] These recommended practices were accepted as tentative by the Society and appear in the A.S.T.M. Methods of Chemical Analysis of Metals (1943), p. 1.

ACTIVITIES OF DIVISIONS AND SUBCOMMITTEES

Division A on Ferrous Metals (Arba Thomas, chairman).—Work has been continued by this division on the study of methods for the determination of aluminum in alloy steels. Plans have been developed to study the combustion method for the determination of sulfur in steels and for small amounts of boron in steels and irons.

The chairman of this division has assisted in the editorial work on the chemical methods in making a detailed review of the Standard Methods of Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 – 42).

T. R. Cunningham, chairman of Subcommittee A-1 on Ferro-Alloys and Subcommittee A-2 on Metallic Materials for Electrical Heating, has reviewed in detail the several methods for ferro-alloys and the newly revised methods for analysis of the electrical-resistance and electrical-heating alloys.

Division B on Non-Ferrous Metals (D. R. Evans, chairman).—Much time and attention has been given by the chairman of this division to plans for the forthcoming edition of the chemical methods and to a review of all the E-3 methods for analysis of non-ferrous metals, as well as to the recommendations on apparatus and reagents.

Subcommittee B-1 on Copper and Its Alloys (R. P. Nevers, chairman) has recommended the withdrawal of three methods for copper alloys, as mentioned earlier in this report. New methods for the analysis of these copper alloys have been prepared,[3] but the subcommittee desires to make a further study of the revised analytical procedures before they are submitted as tentative to Committee E-3.

Subcommittee B-2 on Lead, Tin, Antimony, Bismuth, and Their Alloys (C. A. Ray, chairman) has prepared Proposed Methods for Chemical Analysis of White Metal Bearing Alloys and for Aluminum in Lead- and Tin-Base Solder Metal which will be published as information.[4]

R. W. Bridges, secretary of Subcommittee B-3, has reviewed in detail the Methods for the Analysis of Aluminum and Aluminum Alloys (E 34 – 42 T) and of Magnesium and Magnesium Alloys (E 35 – 42 T).

The revised Methods for Slab Zinc (E 40 – 42 T) and the new Methods for Zinc-Base Die-Casting Alloys (E 47 – 42 T) were reviewed by R. W. Coltman, chairman of Subcommittee B-4. The revised Methods for Chemical Analysis of Nickel (E 39 – 42 T) were reviewed by Calvin Sterling, chairman of Subcommittee B-6.

Division C on Sampling (T. A. Wright, chairman).—Subcommittee 3 on Mechanically Worked Metals under the chairmanship of F. M. Barry has completed a Proposed Method of Sampling Wrought Metals and Alloys for Determination of Chemical Composition.[4]

Division D on General Analytical Methods (B. L. Clarke, chairman).—This division through its Subcommittees on Development, Preparation and Standardization of Reagents (S. E. Q. Ashley, chairman) and on Apparatus (E. B. Rubloff, chairman) prepared the Tentative Recommended Practices for Apparatus and Reagents for Chemical Analysis of Metals[2] being submitted herewith for publication as tentative.

This report has been submitted to letter ballot of the committee which consists of 73 members; 49 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. E. F. LUNDELL.
Chairman.

H. A. BRIGHT,
Secretary.

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[3] See Proposed Methods of Chemical Analysis of Special Brasses and Bronzes, A.S.T.M. Methods of Chemical Analysis of Metals (1943), p. 182.

[4] A.S.T.M. Methods of Chemical Analysis of Metals (1943).

REPORT OF COMMITTEE E-4
ON
METALLOGRAPHY

Committee E-4 on Metallography held a meeting in Atlantic City, N. J., on June 24, 1942, during the Annual Meeting of the Society.

During the past year, Committee E-4 lost two of its most active members through the death of W. H. Swanger, National Bureau of Standards, and of J. F. Wyzalek, Hyatt Roller Bearing Div., General Motors Corp.

The several changes in the membership of the committee have resulted in a net loss of two members, the total membership at this time being 65.

Subsequent to the 1942 Annual Meeting, Committee E-4 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Recommended Practices for:

Identification of Crystalline Materials by the Hanawalt X-ray Diffraction Method (E 43 - 42 T),

Determining the Inclusion Content of Steel (E 45 - 42 T), and

Revision of Tentative Methods of:

Preparation of Metallographic Specimens (E 3 - 39 T).

These recommendations were accepted[1] by Committee E-10 on November 27, 1942, and the new and revised tentative standards appear in the 1942 Book of A.S.T.M. Standards, Part I.

Activities of Subcommittees

Subcommittee I on Selection and Preparation of Samples (G. F. Comstock, chairman).—This subcommittee completed its current work with the development and publication of revised Tentative Methods E 3 - 42 T.

The situation concerning metallographic polishing cloth became critical during the year, and through the efforts of this subcommittee, another source of suitable material has been established.

Subcommittee IV on Photography (L. V. Foster, chairman).—This subcommittee is responsible for the Tentative Methods of Preparation of Micrographs of Metals and Alloys (Including Recommended Practice for Photography as Applied to Metallography) (E 2 - 39 T) which is being continued as tentative.

Subcommittee VI on X-ray Methods (W. L. Fink, chairman).—The Tentative Recommended Practice for Identification of Crystalline Materials by the Hanawalt X-ray Diffraction Method (E 43 - 42 T) was completed and published during the year.

This subcommittee has prepared a Recommended Practice for Analysis of Preferred Orientations in Metals which is the subject of letter ballot in the committee. It is planned to submit this recommended practice to the Society for publication as tentative during the coming year.

A paper on "Preferred Orientation in Rolled Metals; Construction of Pole Figures," by Beulah Field Decker, sponsored by Committee E-4, is being presented at this Annual Meeting of the Society.[2]

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee E-4 reported the following results of the letter ballot vote of a total of 37 ballots returned from a membership of 67: E 43, affirmative 32, negative 0, ballots marked "not voting" 5; E 45, affirmative 35, negative 0, ballots marked "not voting" 2; E 3, affirmative 34, negative 0, ballots marked "not voting" 3.

Through the activities of this subcommittee and the Joint Committee on Chemical Analysis by X-ray Diffraction Methods, a 4000-card index of X-ray diffraction data, for use with the Hanawalt method of chemical analysis by X-ray diffraction (E 43 - 42 T), has been issued by the Society.

Subcommittee VIII on Grain Size (M. A. Grossman, chairman).—No adverse criticism of any of the existing methods for determining grain size have been called to the attention of the subcommittee, and accordingly no revisions are recommended in the existing tentative standards.

Lack of available time has prevented the preparation of recommendations for grain size determination on ferritic materials.

Subcommittee IX on Inclusions (Samuel Epstein, chairman).—This subcommittee, after several years of diligent and conscientious effort, completed the Tentative Recommended Practice for Determining the Inclusion Content of Steel (E 45 - 42 T).

This has been a tremendous undertaking, and the subcommittee is to be congratulated for having brought this matter to a successful conclusion.

This report has been submitted to letter ballot of the committee which consists of 64 members; 37 members returned their ballots, of whom 34 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

J. J. BOWMAN,
Secretary.

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[2] See p. 785.

REPORT OF COMMITTEE E-8
ON
NOMENCLATURE AND DEFINITIONS

The Advisory Committee of Committee E-8 on Nomenclature and Definitions held a meeting in Atlantic City, N. J., on June 23, 1942, during the Annual Meeting of the Society and upon the recommendation of this committee, a number of matters have been handled by correspondence.

The committee has reviewed by correspondence during the year the following definitions prepared by standing committees of the Society:

Tentative Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 42 T), being new definitions prepared by Committee D-1 on Paint, Varnish, Lacquer, and Related Products,

Tentative Method of Test for Spectral Characteristics and Color of Objects and Materials (D 307 - 42 T), prepared by Committee D-1, which includes descriptions of terms and symbols, and

Tentative Definitions of Terms Relating to Soaps and Other Detergents (D 459 - 42 T), prepared by Committee D-12 on Soaps and Other Detergents.

This resulted in the receipt of a number of comments and suggestions respecting the various definitions which have been brought to the attention of the responsible standing committees. Advice has been received from Committee D-12 that the suggestions respecting Definitions D 459 were considered at a meeting held on June 8, 1943, and action taken to revise a number of the definitions. It is expected that the revised definitions will be presented by Committee D-12 to the Society at this annual meeting.[1] The comments with respect to the paint definitions and symbols are still under consideration by Committee D-1.

The proposed new and revised definitions referred to Committee E-8 last year by Committee D-4 on Road and Paving Materials and by Committee D-8 on Bituminous Waterproofing and Roofing Materials have also been reviewed during the year. Various comments on these definitions have been brought to the attention of Committees D-4 and D-8.

The following definitions will shortly be reviewed by Committee E-8:

Tentative Definitions of Terms Relating to Concrete and Concrete Aggregates (C 125 - 42 T), being new and revised definitions prepared by Committee C-9 on Concrete and Concrete Aggregates,

Tentative Descriptive Nomenclature of Objects Made from Plastics (D 675 - 43 T), prepared by Committee D-20 on Plastics, and

Tentative Recommended Practice for Repeated Flexural Stress (Fatigue) Test of Plastics (D 671 - 42 T), prepared by Committee D-20, which includes definitions.

Committee D-13 on Textile Materials is presenting to the Society at this annual meeting several new and revised definitions relating to textile materials. These will also be reviewed by Committee E-8 during the year.

ADOPTION OF TENTATIVE STANDARD AS STANDARD

Subsequent to the 1942 Annual Meeting, Committee E-8 presented to the Society, through Committee E-10 on Standards, proposed Tentative Definitions of Terms Relating to Heat Treatment of Metals. These definitions were accepted[2] by Committee E-10 for publication as tentative on August 24, 1942, to replace immediately the Standard Definitions of Terms Relating to Heat Treatment Operations (Especially as Related to Ferrous Alloys) (A 119 – 33). These tentative definitions were assigned the designation E 44 – 42 T and were published in the 1942 Book of A.S.T.M. Standards, Part I.

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[1] See p. 376.

The definitions were given a detailed review during the year by the Joint Committee on Definitions of Terms Relating to Heat Treatment, comprising representatives of the four sponsoring societies, namely, the American Society for Metals, the Society of Automotive Engineers, the American Foundrymen's Assn., and the American Society for Testing Materials. The revised definitions as approved by the joint committee were referred to the E-8 Subcommittee on Definition of Terms Relating to Heat Treatment (L. H. Fry, chairman) and have been approved by letter ballot of the subcommittee for adoption as standard as follows: From a membership of 9, 8 members returned their ballots all of whom voted affirmatively.

It is accordingly recommended that these revised definitions as appended hereto[3] be approved for reference to letter ballot of the Society for adoption as standard. The results of the vote in Committee E-8 on this recommendation are as follows: From a membership of 52, 38 members returned their ballots, of whom 30 have voted affirmatively, 0 negatively, and 8 members marked their ballots "not voting."

It is recommended that the Tentative Definition of the Term Aggregate (C 58 – 28 T) be continued as tentative without change.

This report has been submitted to letter ballot of the committee which consists of 52 members; 33 members returned their ballots, of whom 26 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

CLOYD M. CHAPMAN,
Chairman.

R. E. HESS,
Ex-officio Secretary.

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[2] In submitting this recommendation to Committee E-10 on Standards, Committee E-8 reported the following results of the letter ballot vote of a total of 36 ballots returned from a committee membership of 52: Affirmative 32, negative 1, and 3 ballots marked "not voting."

[3] These revised definitions were adopted as standard and appear in the 1943 Supplement to Bock of A.S.T.M. Standards, Part I, p. 111.

REPORT OF COMMITTEE E-9

ON

RESEARCH

With this report, Committee E-9 on Research reviews briefly certain research activities and matters related thereto which have engaged its attention during the past year. As customary, a more comprehensive review of research activities throughout the Society will appear in a subsequent issue of the ASTM BULLETIN.[1]

Research Committee Activities:

The Research Committee on Fatigue of Metals has been continuing the study of effect of size on the fatigue strength of notched specimens of two plain carbon steels (SAE 1020 and SAE 1035) and on one heat-treated chromium-molybdenum steel (SAE X4130). The results of these tests have been correlated with the results of tests of unnotched specimens of the same steels reported last year, and a second progress report by H. F. Moore and D. Morkovin on the investigation is being presented at the Annual Meeting.[2]

The Joint Research Committee on Effect of Temperature on the Properties of Metals of The American Society of Mechanical Engineers and this Society is presenting a report[3] at the annual meeting briefly summarizing the progress made during the year in its various research projects. These include tests of tubular members subject to internal pressures, relaxation tests, effect of variables on the high-temperature properties of metals, and comparison of short-time test methods.

Joint Research Committee on Boiler Feedwater Studies.—The experimental work that has been carried on in the joint research committee for the past six years on caustic embrittlement has been terminated and a final report on that work is being prepared in the form of a Bureau of Mines Bulletin to be issued sometime during the current year. Under the auspices of the Joint Research Committee it is planned that the Bureau will serve as a central point for reporting all cases of suspected caustic embrittlement in order that investigators in the future will have a comprehensive and comparable set of data from which to study the industrial occurrence of this phenomenon.

The Joint Research Committee has under consideration a project on silica and turbine blade deposits and is engaged in studying the methods of approach to this new problem.

Joint Research Project on Durability of Concrete.—Due to lack of personnel and inability to secure equipment, it has been found difficult to make much progress on this research project, but it is believed that with the slacking off of activities in the construction field it may be possible to make a little more progress during the next few months.

Proposed Research Committee on Impact Properties of Metals.—Discussions in Committee E-9 on several occasions have led to a proposal to the Executive Committee of the Society that there be formed a Research Committee on Impact Properties of Metals. In making this proposal Committee E-9 has in mind that the Research Committee would not deal with methods of testing (which is in the province of Committee E-1 on Methods of Testing) but would deal with fundamentals of behavior of metals and elements of structure upon impact loading. The organization of this proposed research committee has been authorized by the Executive Committee but its formal organization has been delayed due to pressure of more urgent matters. It is hoped, however, to proceed with the formation of this research committee at an early date.

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[1] ASTM BULLETIN, No. 125, December, 1943, p. 5.
[2] See p. 109.
[3] See p. 125.

RESEARCH PROGRESS OF A.S.T.M. COMMITTEES

A detailed review of research projects carried out by various A.S.T.M. committees is now being made by the Society's staff, from the records contained in the 1942 and 1943 reports of the standing committees. As noted above, a report on this review will be published in the ASTM BULLETIN.[1]

Committee E-9 calls attention to developments in a few of these projects.

Research Activities in Corrosion.—Committee A-5 on Corrosion of Iron and Steel is presenting a report of its Wire Inspection Committee on Field Tests of Wire and Wire Products[4] covering 1941 and 1942 inspections and giving results of atmospheric corrosion tests on specimens of these products after exposure for about six years at each of eleven locations.

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys is reporting on results of tests of 24 non-ferrous metals exposed to atmospheric corrosion for a period of ten years,[5] having previously reported the results of tests at the end of one, three, and six years.

The Advisory Committee on Corrosion, organized a year ago on the recommendation of Committee E-9, has taken hold of its problems in an effective manner and will make possible a better coordination of all of the Society's research corrosion problems. The scope of the committee has been enlarged to include work of interest to Committees B-7 on Light Metals and Alloys, D-1 on Paint, Varnish, Lacquer and Related Products, and D-19 on Water for Industrial Uses, which have accepted invitations to be represented on the Advisory Committee.

Study to Determine if Results of Empirical Flow Tests can be Expressed in Absolute Units.—Studies on this subject initiated two years ago have been completed and a progress report thereon is being presented to the appropriate technical committee of Committee E-1 on Methods of Testing. It is expected that a technical paper describing this work will be available for presentation at the 1944 annual meeting of the Society.

Study of Test Methods for Plastics.—Committee E-9 has been pleased to note that Committee D-20 on Plastics has organized a Subcommittee on Research of Plastics—a noteworthy development in view of the great importance of plastics at this time. The subcommittee has started work on its first project, namely, strength under impact, involving all phases of impact testing.

Synthetic Detergents.—The current report of Committee D-12 on Soaps and Other Detergents, through a paper by J. B. Crowe,[6] gives the results of cooperative studies on a laboratory method for evaluating synthetic detergents.

Research Activities of Other Societies:

The Society has continued its representation on such groups as the Division of Engineering and Industrial Research and the Highway Research Board of the National Research Council, the A.S.M.E.

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[4] See p. 78.

[5] See p. 137.

[6] See p. 378.

Research Committee on Mechanical Springs, and the Welding Research Council of Engineering Foundation. Research activities have been continued along the lines mentioned in previous reports and have been reviewed during the year by Committee E-9.

ADMINISTRATIVE MATTERS

The term of C. E. MacQuigg expires with this Annual Meeting. The appointment of a new member for the term of five years will be made by the Executive Committee of the Society.

The principal of the A.S.T.M. Research Fund is now $34,442.12, an increase of $2050.97, the latter figure comprising $2100 from 1942 and 1943 entrance fees, less $49.03 net loss on sale of securities.

The balance of income in the Research Fund on May 31, 1943, was $1308.22. The present annual available income from the principal of the Fund is about $1140.

Respectfully submitted on behalf of the committee,

G. F. JENKS,
Chairman.

C. L. WARWICK,
Secretary.

June, 1943.

REPORT OF COMMITTEE E-10
ON
STANDARDS

Almost every year seems to bring an increase in the number of recommendations that are submitted to Committee E-10 on Standards for action. This is partly due to the present emergency conditions which account for quite a number of recommendations coming to Committee E-10 from the standing committees. Under the emergency procedure[1] established by the Executive Committee, all recommendations covering the setting up of emergency alternate provisions to A.S.T.M. standards or covering the issuing of complete emergency standards must be approved by Committee E-10. Aside from these recommendations under the emergency procedures, however, the standing committees have submitted to Committee E-10 quite a few recommendations with respect to the publication of new tentative standards, revisions of tentative standards, or tentative revisions of standards.

Only one meeting of the committee was held during the year. However, a procedure was set up whereby recommendations submitted to the committee might be handled by correspondence and letter ballot, and in this way expeditious action has been possible with respect to all matters submitted to Committee E-10. All credit is due its members; although consisting of individuals who are quite burdened, as are most technical men in these times, with more than their regular duties, each recommendation submitted to the committee was carefully scrutinized and a great deal of time and effort was put into the work of the committee.

In addition to the many recommendations acted upon by Committee E-10, this committee has also had before it several proposals for organizing new committee work as referred to later in the report. Although times such as the present would not appear to be particularly opportune for the organization of new committees, nevertheless the several proposals have an urgency which suggests immediate consideration being given to them.

The meeting of Committee E-10 on Standards referred to above was held in Philadelphia, Pa., on August 24, 1942. At that meeting J. R. Townsend was re-elected chairman of Committee E-10 to serve until June, 1943, at which time his term of office as a member of Committee E-10 expires, and R. E. Hess was designated as ex-officio secretary, succeeding C. L. Warwick. The membership on Committee E-10 of F. E. Richart and H. S. Vassar expired last year. The Executive Committee reappointed Messrs. Richart and Vassar to serve as members of Committee E-10 for a three-year term, expiring in 1945.

Review of A.S.T.M. Standardization Activities

A summary of the new and revised tentative standards submitted at the

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[1] The complete emergency procedure is set forth in the 1942 A.S.T.M. Year Book, p. 334.

1942 Annual Meeting was included in the August issue of the ASTM BULLETIN together with a review of the standardization work of the various committees, with statements concerning new standards in prospect. Much of the information, particularly with respect to standardization projects under development in committees, is furnished by the standing committees subsequent to the annual meeting and takes advantage of the fact that many committees plan their next year's work at committee meetings held during the annual meeting.

At the present writing, it is possible to make only a preliminary estimate in terms of new and revised standards of the volume of standardization work accomplished during the year and reported to the Society at this meeting. The reports of standing committees to be presented at the annual meeting include recommendations to the Society substantially as follows:

New tentative standards	45
Revisions of tentative standards	28
Tentative revisions of standards	9
Revisions of standards recommended for adoption as standard	12
Standards and tentative standards withdrawn or replaced	1

There are at present 689 standards and 418 tentative standards of the Society, making a total of 1107, and if the above recommendations are approved this total will become 1151.

CONSIDERATION OF NEW AND REVISED STANDARDS

Proposed new standards accepted by Committee E-10 and published as tentative during the year are as follows:

Tentative Specifications for:

Heat-Treated Carbon-Steel Bolting Material (A 261 - 43 T), submitted by Committee A-1 on Steel,

Copper-Base (Brass) Alloy Die Castings (B 176 - 42 T), submitted by Committee B-6 on Die-Cast Metals and Alloys,

Special Grade Zinc-Base Alloy Die Castings (B 186 - 42 T), submitted by Committee B-6,

Air-Setting Refractory Mortars (Wet Type) for Boiler Furnace and Incinerator Service (C 178 - 43 T), submitted by Committee C-8 on Refractories,

Isopropyl Acetate (D 657 - 42 T), submitted by Committee D-1 on Paint, Varnish, Lacquer, and Related Products,

Crushed Stone, Crushed Slag, and Gravel for Bituminous Concrete Base and Surface Courses of Pavements (D 692 - 42 T), submitted by Committee D-4 on Road and Paving Materials,

Crushed Stone and Crushed Slag for Bituminous Macadam Base and Surface Courses of Pavements (D 693 - 42 T), submitted by Committee D-4,

Crushed Stone, Crushed Slag, and Gravel for Water-Bound Macadam Base and Surface Courses of Pavements (D 694 - 42 T), submitted by Committee D-4,

Asphalt Siding Surfaced with Coarse Mineral Granules (D 699 - 42 T), submitted by Committee D-8 on Bituminous Waterproofing and Roofing Materials,

Phenolic Molding Compounds (D 700 - 43 T), submitted jointly by Committee D-9 on Electrical Insulating Materials and Committee D-20 on Plastics,

Cellulose Nitrate (Pyroxylin) Plastic Sheets, Rods, and Tubes (D 701 - 43 T), submitted by Committee D-20,

Cast Methacrylate Plastic (D 702 - 43 T), submitted by Committee D-20,

Polystyrene Molding Compound (D 703 - 43 T), submitted by Committee D-20,

Melamine-Formaldehyde Molding Compounds (D 704 - 43 T), submitted by Committee D-20,

Urea-Formaldehyde Molding Compounds (D 705 - 43 T), submitted by Committee D-20,

Cellulose Acetate Molding Compounds (D 706 - 43 T) submitted by Committee D-20,

Cellulose Acetate Butyrate Molding Compounds (D 707 - 43 T), submitted by Committee D-20,

Rigid Sheets Made from Vinyl Chloride-Acetate Resins (D 708 - 43 T), submitted by Committee D-20,

Laminated Thermosetting Materials (D 709 - 43 T), submitted jointly by Committee D-9 on Electrical Insulating Materials and Committee D-20, Vulcanized Fibre (D 710 – 43 T), submitted by Committee D-9,

Tentative Methods of:

Test for Permeability and Core Loss of Flat-Rolled Magnetic Materials Using 28-cm. Specimen (A 257 – 42 T), submitted by Committee A-6 on Magnetic Properties,

Test for Incremental Permeability and Core Loss of Flat-Rolled Magnetic Materials at Low Alternating Inductions using 28-cm. Specimen (A 258 – 42 T), submitted by Committee A-6,

Test for Permeability of Feebly Magnetic Materials (A 259 – 42 T), submitted by Committee A-6,

Test for Initial Rate of Absorption (Suction) and Efflorescence of Brick (C 67 – 42 T), submitted by Committee C-15 on Manufactured Masonry Units,

Test for Sieve Analysis and Water Content of Refractory Materials (C 92 – 43 T), submitted by Committee C-8 on Refractories,

Test for Combined Drying and Firing Shrinkage of Fireclay Plastic Refractories (C 179 – 43 T), submitted by Committee C-8,

Panel Test for Resistance to Thermal and Structural Spalling of Fireclay Plastic Refractories (C 180 – 43 T), submitted by Committee C-8,

Test for Workability Index of Fireclay Plastic Refractories (C 181 – 43 T), submitted by Committee C-8,

Test for Thermal Conductivity of Insulating Refractories (C 182 – 43 T), submitted by Committee C-8,

Evaluating Degree of Chalking of Exterior Paints of the Linseed-Oil Type (D 659 – 42 T), submitted by Committee D-1 on Paint, Varnish, Lacquer, and Related Products,

Evaluating Degree of Checking of Exterior Paints of the Linseed-Oil Type (D 660 – 42 T), submitted by Committee D-1,

Evaluating Degree of Cracking of Exterior Paints of the Linseed-Oil Type (D 661 – 42 T), submitted by Committee D-1,

Evaluating Degree of Erosion of Exterior Paints of the Linseed-Oil Type (D 662 – 42 T), submitted by Committee D-1,

Test for Compressive Strength of Plastics (D 695 – 42 T), submitted by Committee D-20 on Plastics,

Test for Coefficient of Linear Thermal Expansion of Plastics (D 696 – 42 T), submitted by Committee D-20,

Test for Water Vapor Permeability of Plastic Sheets (D 697 – 42 T), submitted by Committee D-20,

Test for Moisture-Density Relations of Soils (D 698 – 42 T), submitted jointly by Committee D-4 on Road and Paving Materials and Committee D-18 on Soils for Engineering Purposes,

Chemical Analysis for Selenium in Steel (E 30 – 43 T), submitted by Committee E-3 on Chemical Analysis of Metals,

Chemical Analysis of Lead- and Tin-Base Solder Metal (E 46 – 42 T), submitted by Committee E-3,

Chemical Analysis of Zinc-Base Die Casting Alloys (E 47 – 42 T), submitted by Committee E-3,

Tentative Recommended Practices for:

Torsion Tests of Cast Iron (A 260 – 42T), submitted by Committee A-3 on Cast Iron,

Chromium Plating on Steel for Engineering Use (B 177 – 43 T), submitted by Committee B-8 on Electrodeposited Metallic Coatings,

Characteristics of Standard Carbon Arc Accelerated Weathering Unit (E 42 – 42 T), submitted by Committee E-1 on Methods of Testing,

Identification of Crystalline Materials by the Hanawalt X-ray Diffraction Method (E 43 – 42 T), submitted by Committee E-4 on Metallography,

Determining the Inclusion Content of Steel (E 45 – 42 T), submitted by Committee E-4,

Tentative Definition of:

Terms Relating to Conditioning and Weathering with Procedures (E 41 – 42 T), submitted by Committee E-1 on Methods of Testing, and

Terms Relating to Heat-Treatment of Metals (E 44 – 42 T).

Revisions of the following tentative standards and tentative revisions of standards were accepted by Committee E-10:

Tentative Specifications for:

Zinc Coating (Hot-Dip) on Iron and Steel Hardware (A 153 – 42 T), submitted by Committee A-5 on Corrosion of Iron and Steel,

Cartridge Brass Sheet, Strip, and Disks (B 19 – 42 T), submitted by Committee B-5 on Copper and Copper Alloys, Cast and Wrought,

Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 – 42 T), submitted by Committee B-5,

Brass Sheet and Strip (B 36 – 42 T), submitted by Committee B-5, Leaded Brass Sheet and Strip (B 121 – 42 T), submitted by Committee B-5,

Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 – 42 T), submitted by Committee B-5,

Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 42 T), submitted by Committee B-5,

Cartridge Brass Cartridge Case Cups (B 129 – 42 T), submitted by Committee B-5,

Gilding Metal Sheet and Strip (B 130 – 42 T), submitted by Committee B-5,

Gilding Metal Bullet Jacket Cups (B 131 – 42 T), submitted by Committee B-5,

Miscellaneous Brass Tubes, (B 135 – 42 T), submitted by Committee B-5,

Phosphor Bronze Rods, Bars, and Shapes (B 139 – 42 T), submitted by Committee B-5,

Aluminum-Bronze Sand Castings (B 148 – 42 T), submitted by Committee B-5,

Phosphor Bronze Wire (B 159 – 42 T), submitted by Committee B-5,

Aluminum Bronze Sheet and Strip (B 169 – 42 T), submitted by Committee B-5,

Copper-Alloy Condenser Tube Plates (B 171 – 42 T), submitted by Committee B-5,

Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 – 42 T), submitted by Committee D-8 on Bituminous Waterproofing and Roofing Materials,

Standard Sizes of Coarse Aggregate for Highway Construction (D 448 – 42 T), submitted by Committee D-4 on Road and Paving Materials,

A.S.T.M. Thermometers (E 1 – 43 T), submitted by Committee E-1 on Methods of Testing,

Tentative Methods of:

Testing Molded Materials Used for Electrical Insulation (D 48 – 42 T), submitted by Committee D-9 on Electrical Insulating Materials,

Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75 – 42 T), submitted by Committee D-4,

Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 – 42 T), submitted by Committee D-9 on Electrical Insulating Materials,

Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 – 42 T), submitted on Committee D-9,

Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (D 228 – 42 T), submitted by Committee D-8 on Bituminous Waterproofing and Roofing Materials,

Surveying and Sampling Soils for Highway Subgrades (D 420 – 42 T), submitted by Committee D-18 on Soils for Engineering Purposes,

Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 – 43 T), submitted by Committee D-11 on Rubber Products.

Test for Tensile Properties of Plastics (D 638 – 42 T), submitted by Committee D-20 on Plastics,

Preparation of Metallographic Specimens (E 3 – 42 T), submitted by Committee E-4 on Metallography,

Test for Softening Point (Ball and Shouldered Ring Apparatus) (E 28 – 42 T), submitted by Committee E-1 on Methods of Testing,

Tentative Recommended Practice for:

Short-Time Elevated Temperature Tension Tests of Metallic Materials (E 21 – 42 T), submitted by Joint A.S.M.E.-A.S.T.M. Research Committee on Effect of Temperature on the Properties of Metals,

Tentative Classification of:

Terms and Descriptive Nomenclature of Objects Made from Plastics (D 675 – 43 T), submitted by Committee D-20 on Plastics,

Standard Specifications for:

Hollow Loading-Bearing Concrete Masonry Units (C 90 – 39), submitted by Committee C-15 on Manufactured Masonry Units,

Zinc Oxide (D 79 – 41), submitted by Committee D-1 on Paint, Varnish, Lacquer, and Related Products,

Standard Methods of:

Test for Pyrometric Cone Equivalent of Refractory Materials (C 24 – 42), submitted by Committee C-8 on Refractories,

Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (C 25 – 29), submitted by Committee C-7 on Lime,

Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (C 38 – 42), submitted by Committee C-8,

Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 – 42), submitted by Committee C-8,

Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 – 42), submitted by Committee C-8,

Mechanical Analysis of Soils (D 422 – 39), submitted by Committee D-18 on Soils for Engineering Purposes, and

Standard Definitions of:

Terms Relating to Refractories (C 71 – 42), submitted by Committee C-8.

The withdrawal of the following standards and tentative standards was approved by Committee E-10:

Standard Specifications for:

Seamless Cold-Drawn Alloy-Steel (4 to 6 per cent Chromium) Heat-Exchanger and Condenser Tubes (A 187 – 40), recommended by Committee A-1 on Steel,

Seamless Alloy-Steel (4 to 6 per cent Chromium) Still Tubes for Refinery Service (A 188 – 40), recommended by Committee A-1,

Curing Portland-Cement Concrete (C 80 – 34), recommended by Committee C-9 on Concrete and Concrete Aggregates,

Gravel for Bituminous Concrete Base (D 309 – 30), recommended by Committee D-4 on Road and Paving Materials,

Standard Methods of:

Curing Portland-Cement Concrete Slabs with Bituminous Coverings (C 81 – 36), recommended by Committee C-9,

Curing Portland-Cement Concrete with Calcium Chloride Admixture (C 82 – 38), recommended by Committee C-9,

Curing Portland-Cement Concrete Slabs by Surface Application of Calcium Chloride (C 83 – 38), recommended by Committee C-9,

Curing Portland-Cement Concrete Slabs with Wet Coverings (C 84 – 36), recommended by Committee C-9,

Test for Particle Size of Ground Refractory Materials (C 92 – 36), recommended by Committee C-8 on Refractories,

Standard Definitions of:

Terms Relating to Heat Treatment Operations (Especially as Related to Ferrous Alloys) (A 119 – 33), recommended by Committee E-8 on Nomenclature and Definitions,

Tentative Specifications for:

Crushed Stone for Bituminous Macadam Base and Surface Courses (D 192 – 41 T), recommended by Committee D-4 on Road and Paving Materials,

Crushed Slag for Bituminous Concrete Base and Surface Courses (D 485 – 38 T), recommended by Committee D-4,

Crushed Stone for Bituminous Concrete Base and Surface Courses (D 486 – 39 T), recommended by Committee D-4,

Crushed Slag for Bituminous Macadam Base and Surface Courses (D 487 – 42 T), recommended by Committee D-4,

Crushed Slag for Water-Bound Base and Surface Courses (D 488 – 38 T), recommended by Committee D-4,

Crushed Stone for Water-Bound Base and Surface Courses (D 489 – 40 T), recommended by Committee D-4,

Tentative Methods of:

Chemical Analysis of Alloys of Lead, Tin, Antimony, and Copper (B 18 – 36 T), recommended by Committee E-3 on Chemical Analysis of Metals,

Chemical Analysis of Manganese Bronze (B 27 – 36 T), recommended by Committee E-3,

Chemical Analysis of Gun Metal (B 28 – 36 T), recommended by Committee E-3, and

Chemical Analysis of Bronze Bearing Metal (B 46 – 36 T).

ACTIONS UNDER EMERGENCY PROCEDURE

Emergency Alternate Provisions.—A large number of emergency alternate provisions have been accepted by Committee E-10 during the year as follows:

Designation	Emergency Alternate Provisions in	Committee
	Standard Specifications for:	
EA – A 21	Carbon-Steel Axles for Cars and Tenders (A 21 – 36)	A-1
EA – A 25a	Wrought Steel Wheels for Electric Railway Service (A 25 – 41)	A-1
EA – A 53	Welded and Seamless Steel Pipe (A 53 – 42)	A-1
EA – A 57	Multiple-Wear Wrought Steel Wheels (A 57 – 39)	A-1
EA – A 83	Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 42)	A-1
EA – A 120	Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 42)	A-1
EA – A 134	Electric-Fusion-Welded Steel Pipe (Sizes 30 in. and Over (A 134 – 42)	A-1
EA – A 135	Electric-Resistance-Welded Steel Pipe (A 135 – 42)	A-1
EA – A 139a	Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 – 42)	A-1
EA – A 148a	Alloy-Steel Castings for Structural Purposes (A 148 – 42)	A-1
EA – A 161	Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 – 40)	A-1
EA – A 178	Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 40)	A-1
EA – A 179	Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 42)	A-1
EA – A 186	One-Wear and Two-Wear Wrought Steel Wheels (A 186 – 39)	A-1
EA – A 190	Lightweight and Thin-Sectioned Gray-Iron Castings (A 190 – 40)	A-3

EA - A 192	Seamless Steel Boiler Tubes for High-Pressure Service (A 192 - 40)	A-1
EA - A 199	Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 - 40)	A-1
EA - A 200	Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 - 40)	A-1
EA - A 209	Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 - 42)	A-1
EA - A 211	Spiral-Welded Steel or Iron Pipe (A 211 - 40)	A-1
EA - A 213	Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 - 42)	A-1
EA - A 214	Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 - 42)	A-1
EA - A 226	Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 40)	A-1
EA - A 235	Carbon-Steel Forgings for General Industrial Use (A 235 - 42)	A-1
EA - A 236a	Carbon-Steel Forgings for Locomotives and Cars (A 236 - 42)	A-1
EA - A 237	Alloy-Steel Forgings for General Industrial Use (A 237 - 42)	A-1
EA - A 238	Alloy-Steel Forgings for Locomotives and Cars (A 238 - 42)	A-1
EA - A 244	Heat-Treated Wrought Steel Wheels (A 244 - 42)	A-1
EA - A 249	Atomic-Hydrogen-Arc-Welded and Electric-Resistance-Welded Alloy-Steel Boiler and Superheater Tubes (A 249 - 42)	A-1
EA - B 8	Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 - 41)	B-1
EA - B 12	Copper Bars for Locomotive Staybolts (B 12 - 42)	B-5
EA - B 23a	White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (B 23 - 26)	B-2
EA - B 85a	Aluminum-Base Alloy Die Castings (B 85 - 42)	B-6
EA - B 100	Rolled Copper-Alloy Bearing and Expansion Plates for Bridge and Other Structural Uses (B 100 - 40)	B-5
EA - B 103	Phosphor Bronze Sheet and Strip (B 103 - 42)	B-5
EA - B 111	Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 42)	B-5
EA - D 69	Friction Tape for General Use for Electrical Purposes (D 69 - 38)	D-11
EA - D 119	Rubber Insulating Tape (D 119 - 38)	D-11
EA - D 375	Asbestos Roving for Electrical Purposes, and Methods of Test (D 375 - 42)	D-13
EA - D 469a	Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 - 41)	D-11
EA - D 607	Mica Pigment (D 607 - 42)	D-1
	Tentative Specifications for:	
EA - A 158	Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 - 42 T)	A-1
EA - A 206	Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 - 42 T)	A-1
EA - A 250	Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 - 41 T)	A-1
EA - B 19	Cartridge Brass Sheet, Strip, and Disks (B 19 - 42 T)	B-5
EA - B 22	Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 - 40 T)	B-5
EA - B 32a	Soft Solder Metal (B 32 - 40 T)	B-2
EA - B 36	Brass Sheet and Strip (B 36 - 41 T)	B-5
EA - B 86a	Zinc-Base Alloy Die Castings (B 86 - 41 T)	B-6
EA - B 121	Leaded Brass Sheet and Strip (B 121 - 41 T)	B-5
EA - B 122	Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 - 39 T)	B-5
EA - B 124	Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 - 42 T)	B-5
EA - B 129	Cartridge Brass Cartridge Case Cups (B 129 - 40 T)	B-5
EA - B 130	Gilding Metal Sheet and Strip (B 130 - 41 T)	B-5
EA - B 131	Gilding Metal Bullet Jacket Cups (B 131 - 40 T)	B-5
EA - B 133	Copper Rods, Bars, and Shapes (B 133 - 42 T)	B-5
EA - B 134	Brass Wire (B 134 - 42 T)	B-5
EA - B 135	Miscellaneous Brass Tubes (B 135 - 42 T)	B-5
EA - B 139	Phosphor Bronze Rods, Bars, and Shapes (B 139 - 42 T)	B-5
EA - B 148a	Aluminum-Bronze Sand Castings (B 148 - 42 T)	B-5
EA - B 151	Copper-Nickel-Zinc Alloy Rod and Wire (B 151 - 42 T)	B-5
EA - B 152	Copper Sheet, Strip, and Plate (B 152 - 41 T)	B-5
EA - B 159	Phosphor Bronze Wire (B 159 - 42 T)	B-5
EA - B 169	Aluminum Bronze Sheet and Strip (B 169 - 41 T)	B-5
EA - B 171	Copper-Alloy Condenser Tube Plates (B 171 - 42 T)	B-5
EA - D 532a	Rubber Sheath Compound for Electrical Insulated Cords and Cables (D 532 - 39 T)	D-11
	Standard Method of:	
EA - D 524	Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 - 42)	D-2

The withdrawal of the following emergency alternate provisions was approved by Committee E-10:

Designation	Emergency Alternate Provisions in	Committee
	Standard Specifications for:	
EA - D 82	Basic Sulfate White Lead (D 82 - 41)	D-1
EA - D 496	Chip Soap (D 496 - 39)	D-12
EA - D 498	Powdered Soap (Nonalkaline Soap Powder) (D 498 - 39)	D-12
	Tentative Specifications for:	
EA - B 158	Rope-Lay-Stranded and Bunch-Stranded Copper Cables for Electrical Conductors (B 158 - 41 T)	B-1

Emergency Standards.—Twenty five emergency standards have been accepted by Committee E-10 during the year as follows:

Emergency Specifications for:

Carbon-Steel and Alloy-Steel Forgings for Magnetic Retaining Rings for Turbine Generators (ES - 21), submitted by Committee A-1 on Steel,

Alloy-Steel Forgings for Nonmagnetic Coil Retaining Rings for Turbine Generators (ES - 22), submitted by Committee A-1,

Carbon-Steel Forgings for Rings for Main Reduction Gears (ES - 23), submitted by Committee A-1,

Carbon-Steel and Alloy-Steel Forgings for Pinions for Main Reduction Gears (ES – 24) submitted by Committee A-1,
Carbon-Steel and Alloy-Steel Forgings for Turbine Generator Rotors and Shafts (ES – 25), submitted by Committee A-1,
Carbon-Steel and Alloy-Steel Forgings for Turbine Rotors and Shafts (ES – 26), submitted by Committee A-1,
Carbon-Steel and Alloy-Steel Forgings for Turbine Bucket Wheels (ES – 27), submitted by Committee A-1,
Malleable Iron Flanges, Pipe Fittings, and Valve Parts (ES – 20), submitted by Committee A-7,
Fire-Refined Copper for Wrought Products and Alloys (ES – 7), submitted by Committee B-2 on Non-Ferrous Metals and Alloys,
Special Quality Aluminum-Base Alloy Die Castings (ES – 29), submitted by Committee B-6 on Die-Cast Metals and Alloys,
Electrodeposited Coatings of Lead on Steel (ES – 31), submitted by Committee B-8 on Electrodeposited Metallic Coatings,
85 Per Cent Magnesia Thermal Insulating Cement (ES – 8), submitted by Committee C-16 on Thermal Insulating Materials,
Long Fiber Asbestos Thermal Insulating Cement (ES – 9), submitted by Committee C-16,
Mineral Wool Thermal Insulating Cement (ES – 10), submitted by Committee C-16,
Expanded or Exfoliated Mica Thermal Insulating Cement (ES – 11), submitted by Committee C-16,
Diatomaceous Silica Thermal Insulating Cement for Use from 600 to 1200 F. (ES – 12), submitted by Committee C-16,
Diatomaceous Silica Thermal Insulating Cement, for Use from 1200 to 1900 F. (ES – 13), submitted by Committee C-16,
Blanket Thermal Insulation for Building Purposes (ES - 14), submitted by Committee C-16,
Blanket Thermal Insulation for Industrial Purposes (ES – 15), submitted by Committee C-16,
Blanket Thermal Insulation for Refrigeration Purposes (ES – 16), submitted by Committee C-16,
Preformed Pipe Covering Thermal Insulation (ES – 17), submitted by Committee C-16,
Preformed Block Thermal Insulation (ES – 18), submitted by Committee C-16,
Structural Board Insulation (Thermal Insulation) (ES – 19), submitted by Committee C-16,
Chloroprene Sheath Compound for Electrical Insulated Cords and Cables (ES – 28), submitted by Committee D-11 on Rubber Products, and
Chloroprene Sheath Compound for Electrical Insulated Cords and Cables Where Extreme Abrasion Resistance is Not Required (ES – 30), submitted by Committee D-11.

Revisions of the following emergency specifications were accepted by Committee E-10:

Emergency Specifications for:

Lead-Coated and Lead-Alloy-Coated Copper Wire for Electrical Purposes (ES – 1a), submitted by Committee B-1 on Copper and Copper Alloy Wires for Electrical Conductors, and
Carbon-Chromium Ball and Roller-Bearing Steels (ES – 5a), submitted by Committee A-1 on Steel.

The withdrawal of the Emergency Standard Hardness Conversion Table for Cartridge Brass (ES – 4), recommended by Committee E-1 on Methods of Testing, was approved by Committee E-10.

PROMOTION AND EXPANSION OF STANDARDIZATION ACTIVITIES

Certain discussions and developments have taken place in recent months with respect to the organization of new committees in the Society. In line with the recommendations of a special study group, functioning under the chairmanship of R. P. Anderson, Committee E-10 is now recommending to the Executive Committee the formation of a new standing committee on industrial aromatic hydrocarbons to cover nomenclature, methods of test, and specifications for industrial aromatic hydrocarbons and related products boiling below 400 F. for other than fuel purposes.

Largely as a result of the Symposium on Powder Metallurgy held at the time of the Spring Group Meeting of the Society in Buffalo, N. Y., it has been proposed that the Society organize a standing committee on this subject. A study committee, under the chairmanship of J. R. Townsend, is now looking into the question in order to advise the Executive Committee concerning the desirability or otherwise of organizing a standing committee on powder metallurgy at this time.

Reference has been made from time to time in the reports of Committee E-10 of suggestions that work be organized in the field of adhesives. Recently the extensive use of adhesives in the plywood and similar fields has suggested the desirability of proceeding fairly promptly with the organization of a committee on adhesives. M. H. Bigelow, a member of the Executive Committee of the Society, is heading up the study committee now being appointed to explore the field with respect to adhesives.

Some thought is also being given to the proposal that the Society undertake standardization work in the field of porcelain enamels, which question has recently been reopened.

RELATIONS WITH THE AMERICAN STANDARDS ASSOCIATION

Standards Submitted Under Existing Standards and Proprietary Procedure.—The existing standards method for submitting standards to the American Standards Association whereby it is necessary to show that the standard is supported by the necessary consensus of those substantially concerned with its scope and provisions, is particularly appropriate for a large number of A.S.T.M. standards. Under this procedure, the following specifications have been approved during the year by the ASA as American Standard:

Standard Specifications for:

Forged or Rolled Steel Pipe Flanges for General Service (A 181 - 42; ASA G46.1—1942),
Carbon-Steel Castings for Miscellaneous Industrial Uses (A 27 - 42; G50.1—1943),
Carbon-Steel Castings Suitable for Fusion Welding for Miscellaneous Industrial Uses (A 215 - 41; G51.1—1943),
Alloy-Steel Castings for Structural Purposes (A 148 - 42; G52.1—1943),
Slab Zinc (Spelter) (B 6 - 37; H24.1—1943), and
Rolled Zinc (B 69 - 39; H25.1—1943).

The Society has been granted proprietary sponsorship for the revision of standards approved under the existing standards method and, functioning as proprietary sponsor, has submitted to the ASA during the year revisions of the following standards, all of which have been approved:

Standard Specifications for:

Gypsum Lath (C 37 - 42; ASA A67.1—1942),
Gypsum Sheathing Board (C 79 - 42; A68.1—1942),
Gypsum Wall Board (C 36 - 42; A69.1—1942),
Steel for Bridges and Buildings (A 7 - 42; G24—1942),
Carbon-Steel Plates for Stationary Boilers and Other Pressure Vessels (A 70 - 42; G29.1—1942),
Low-Carbon Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 - 42; G33.1—1942),
Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 - 42; G34.1—1942),
Alloy-Steel Castings for Valves, Flanges, and Fittings for Service at Temperatures from 750 to 1100 F. (A 157 - 42; G36.1—1942),
Structural Steel for Locomotives and Cars (A 113 - 42; G39.1—1942),
Forged or Rolled Steel Pipe Flanges for General Service (A 181 - 42; G46.1—1942),
Free-Cutting Brass Rod for Use in Screw Machines (B 16 - 42; H8—1942),
Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 - 42; H17.1—1942),
Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 - 42; H17.2—1942),
Textile Testing Machines (D 76 - 42; L15.1—1943).

Standard Methods of:

Testing Gypsum and Gypsum Products (C 26 - 42; A70.1—1942),
Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572 - 42; J4.1—1943),

Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (D 573 - 42; J5.1—1943), and
Laboratory Sampling and Analysis of Coal and Coke (D 271 - 42; K18—1942).

On the recommendation of the Society functioning as proprietary sponsor, approval as American Standard of the obsolete Tentative Methods of Chemical Analysis of Manganese Bronze (B 27-36 T; K3—1935) has been withdrawn by the ASA.

Approval as American Tentative Standard of the following obsolete specifications and methods has been withdrawn by the ASA on the recommendation of the Society as proprietary sponsor:

Standard Specifications for Solder Metal (B 32 - 21; H11—1924), and
Tentative Methods of Chemical Analysis of Gun Metal (B 28 - 36 T; K4—1921).

Standards Submitted Under Sectional Committee Procedure.—Reports were received from several of the sectional committees for which the Society is sponsor or joint sponsor, submitting standards to the ASA for approval. These reports were transmitted to that association and the following standards have been approved for the first time or reapproved in revised form during the year as American Standard:

On the Recommendation of Sectional Committee A2 on Specifications for Fire Tests of Materials and Construction:

Fire Tests of Building Construction and Materials (C 19 - 41; ASA A2.1—1942), and
Fire Tests of Door Assemblies (C 152 - 41; A2.2—1942).

On the Recommendation of Sectional Committee A37 on Road and Paving Materials:

Test for Determination of Bitumen (D 4 - 42; ASA A37.3—1943),
Test for Amount of Material Finer than No. 200 Sieve in Aggregates (C 117 - 37; A37.4—1943)
Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 - 42; A37.5—1943),
Test for Specific Gravity and Absorption of Fine Aggregate (C 128 - 42; A37.6—1943),
Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 - 39; A37.7—1943),
Sieve Analysis of Fine and Coarse Aggregates (C 136 - 39; A37.8—1943),
Test for Distillation of Tar Products Suitable for Road Treatment (D 20 - 30; A37.9—1943)
Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (D 36 - 26; A37.10—1943),
Test for Ductility of Bituminous Materials (D 113 - 39; A37.11—1943),
Test for Proportion of Bitumen Soluble in Carbon Tetrachloride (D 165 - 42; A37.12—1943),
Test for Residue of Specified Penetrations (D 243 - 36; A37.13—1943), and
Sieve Analysis of Mineral Filler (D 546 - 41; A37.14—1943).

On the Recommendation of Sectional Committee A42 on Specifications for Plastering:

Specifications for Gypsum Plastering, including Requirements for Lathing and Furring (A42.1—1942).

On the Recommendation of Sectional Committee B36 on Standardization of Dimensions and Materials of Wrought-Iron and Wrought-Steel Pipe and Tubing:

Specifications for Welded and Seamless Steel Pipe (A 53 - 42; ASA B36.1—1942),
Specifications for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 42 T; B36.3—1942),
Specifications for Electric-Fusion-Welded Steel Pipe (Sizes 30 in. and Over) (A 134 - 42; B36.4—1942),
Specifications for Electric-Resistance-Welded Steel Pipe (A 135 - 42; B36.5—1942),
Specifications for Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 - 42; B36.9—1942),
Specifications for Electric-Fusion-Welded Steel Pipe for High-Temperature and High-Pressure Service (A 155 - 42; B36.11—1942),
Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 - 42; B36.12—1942),
Specifications for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 - 40; B36.13—1942),
Specifications for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 - 40; B36.14—1942),
Specifications for Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 - 40; B36.15—1942),
Specifications for Spiral-Welded Steel or Iron Pipe (A 211 - 40; B36.16—1942),

- Specifications for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 - 42; B36.17—1942), and
- Specifications for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 40; B36.18—1942)

On the Recommendation of Sectional Committee G8 on Specifications for Zinc Coating of Iron and Steel:

- Specifications for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for ordinary uses (A 120-42; ASA G8.7-1943), and
- Specifications for Zinc-Coated Steel Wire Strand ("Galvanized" and Class A ("Extra Galvanized")) (A 122-41; G8.6-1943).

On the Recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants:

- Test for Melting Point of Paraffin Wax (D 87 - 42; ASA Z11.4—1942),
- Test for Flash Point by Means of the Pensky-Martens Closed Tester (D 93 - 42; Z11.7—1942),
- Test for Gum Content of Gasoline (D 381 - 42; Z11.36—1942),
- Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 42; Z11. 4—1942),
- Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 - 42; Z11.47—1942), and
- Test for Tetraethyl Lead in Gasoline (D 526 - 42 Z11.48—1942).

On the recommendation of Sectional Committee B36 on Standardization of Dimensions and Materials of Wrought-Iron and Wrought-Steel Pipe and Tubing approval as American Standard of the obsolete Specifications for Lock-Bar Steel Pipe (A 137 - 34; B36.7—1935) was withdrawn by the ASA.

ASA WAR COMMITTEE ON RADIO ORGANIZED

At the request of the War Production Board, the War Committee on Radio was formed under the war standards procedure of the American Standards Association to set up war standards for components for military radio to help speed production of radio equipment for the Army and Navy, make replacements of parts easier, and to conserve materials. Thirteen subcommittees have been formed handling specific divisions of this work.

Since A.S.T.M. Committee D-9 on Electrical Insulating Materials is interested in a number of the materials in question, a liaison has been established through the appointment of L. J. Cavanaugh to Subcommittee I of the War Committee on Radio which subcommittee is concerned with insulating materials. This appointment was made in order to avoid a duplication of effort.

Upon the organization under this subcommittee of two new drafting groups (7 and 8) dealing with plastics, arrangements were effected (in view of the vast amount of work on plastics that had been carried out by A.S.T.M. Committee D-20 on Plastics) for the new Drafting Group 7 on Materials Specifications for Plastics to look to Committee D-20, and specifically to Subcommittee VI of that committee, for specifications on plastics rather than to initiate any work on plastics themselves unless it develops that Subcommittee VI is not in a position to take care of it. Messrs. M. P. Davis, W. A. Zinzow, Robert Burns, L. W. A. Meyer, and G. M. Kline were appointed to serve on Task Group 7 and Mr. Zinzow was appointed to serve on Task Group 8 on Product Specifications for Plastic Communications Components. In this way the activities of the task groups are coordinated with those of our Committees D-9 and D-20.

In addition to the work of the War Committee on Radio, other war committees are being appointed by the ASA in order to meet requests from the Office of Price Administration and other Government agencies to establish standards having to do with safety clothing. Here again, there may be certain questions dealing with materials in connection with which the Society may be able to contribute and accordingly the work is being followed very closely. In all these activities, it is recognized that much of the A.S.T.M. work may have a direct bearing and in this connection it may be able to be of considerable assistance. There is a general understanding that with respect to any materials questions the A.S.T.M. will be consulted.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

R. E. HESS,
Ex-officio Secretary.

TECHNICAL PAPERS

With Discussions

EDGAR MARBURG LECTURE

The purpose of the Edgar Marburg Lecture is to have described at the Annual Meetings of the American Society for Testing Materials, by leaders in their respective fields, outstanding developments in the promotion of knowledge of engineering materials. Established as a means of emphasizing the importance of promoting knowledge of materials, the Lecture honors and perpetuates the memory of Edgar Marburg, first Secretary of the Society.

WOOD AS AN ENGINEERING MATERIAL[1]

Eighteenth Edgar Marburg Lecture[2]

By L. J. Markwardt[3]

Introduction

> "This is the forest primeval. The murmuring pines and the hemlocks,
> Bearded with moss, and in garments green, indistinct in the twilight, . . ."
>
> —*Longfellow.*

Wood was among the first structural materials in the primitive world; yet significantly, it still maintains an indispensable place in the modern age. Although the present World War is commonly thought of as one of high-speed fighters and streamlined bombers, dashing torpedo boats and evasive submarines, mechanized transport and armored tanks, there is required to make these war machines function a larger quantity and greater variety of wood products than ever before. In the United States alone the annual lumber production last year was over 32 billion board feet—enough wood to lay an airplane landing mat, 1 in. thick and 240 ft. wide, completely around the world at the equator. More than one third of this vast quantity of lumber is assigned to the task of securely packaging war supplies for shipment to the far corners of the world, and only restricted manpower in the forests limits the production to these indicated levels.

Lumber and other wood products constitute one of the major industrial groups of the country. According to the census figures, the industry employed in 1939 more than one million persons and produced material valued at $4,410-000,000. Among the industries, it ranked fourth in number of wage earners and sixth in value of product.

Trees are the oldest of living things. The age of the celebrated Dragon Tree of Orotavia has been estimated at 6000 yr.; and the so-called Mexican cedar was estimated at 4000 yr. The age of a Sierra juniper in Stanislaus National Forest, Calif., is estimated as at least 6000 yr., while actual age determinations on the California bigtree (*Sequoia gigantea*) by means of a ring count indicated ages of 4000 yr.

The tallest living tree for which authentic measurements are available is reported to be a redwood (*Sequoia sempervirens*) in Humboldt State Redwood Park, Calif., with a height of 364 ft.

Another redwood tree in Humboldt County, Calif., is reported to scale 361,300 board feet of merchantable lumber, enough to build about 20 homes. The famous General Sherman tree in California is estimated to contain 250-000 board feet.

Since wood has been the servant of man from time immemorial, there has naturally accumulated an extensive empirical background regarding its use. As would be expected, some of the "facts" which have come to us in this way bear the earmarks of superstition, but prejudice and rule-of-thumb have been largely dispelled by the extensive and intensive research that has marked the past few decades. Nevertheless, there are still in the popular mind many things presumably known about wood that are not so. These aspects leave ground for some credence in the much quoted platitude that "It is not the things we don't know that do us the most harm, but the ones we do know that are not so." It is not long ago, relatively speaking, that certain specifications called for the felling of trees in a particular phase of the moon, and that kiln drying of lumber, even under scientifically controlled conditions, was looked upon askance. Although wood has a long and varied history which it would be interesting to recount, space precludes even a brief review here.

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[1] Read on June 30, 1943, before the Annual Meeting of the American Society for Testing Materials, Pittsburgh, Pa.

[2] In the preparation of this manuscript, publications of various staff members of the Forest Products Laboratory have been consulted and freely quoted. Special acknowledgment is made to C. P. Winslow, K. F. Wendt, G. M. Hunt, G. J. Ritter, and L. W. Wood for their contributions and assistance.

[3] Chief, Division of Timber Mechanics, Forest Products Laboratory, Forest Service, U. S. Department of Agriculture, maintained at Madison, Wis., in cooperation with the University of Wisconsin.

A Forest of Virgin Redwoods in California.

Trees are the oldest of living things. The age of a Sierra juniper in the Stanislaus National Forest has been estimated at 6000 yr.

Because wood is relatively light in weight and is easy to shape and form, it lends itself readily to transportation and to fabrication even by pioneer methods and tools. Also, while it is an exceedingly complicated material both from the standpoint of physical structure and chemical composition, early craftsmen attained with it a degree of success that was a marked tribute to their ingenuity and to the time.

Where close design was not critical, wood was thus made to serve effectively through the centuries by employing experience and trial-and-error methods, without the critical need of the more intimate knowledge of properties on which many modern applications depend. Thus, in building his famous Ark of gopher wood,[4] Noah did not need to know that he was working with a highly complicated orthotropic material with a wide variety of properties. The same was true through the years to come, when the Egyptians invented plywood, and discovered a way to reorient the material and diversify it further by more nearly equalizing the strength properties; when the Romans streamlined the Ark into galleys, rowed by slaves; and when the Vikings "by oar and mast, not only Greenland did explore, but also America's verdant shore." But when the skilled craftsmen centuries ago constructed the ingenious arches and trusses that support the high vaulted roofs of the great cathedrals of Europe they no doubt recognized joints and fastenings as the "Achilles heel" of wood construction, and yearned for the modern connectors and the fabrication technique this twentieth century has made available.

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[4] Regarded by Celsius to be cedar, but by Chayne to be cypress.

Wood users of the world have a wide choice of species, each of which exhibits, at least in some degree, properties or characteristics that are individual to itself. Around the particular properties of certain of these woods has developed a utility and demand for specific uses which has created a substantial world commerce. Thus, teak (*Tectona grandis*) has long been the favored wood for warship decks; lignum vitae for propeller bearings of ships; mahogany for small boats; and spruce, because of its favorable combination of weight and strength, for various airplane structural elements.

In some respects, each species with different inherent properties may be considered as a different alloy. Yet among the large variety of species are many sufficiently alike in properties to be used interchangeably in closely designed engineering structures.

While the variety of tree species in the world is enormous, the exact number is unknown, as vast forest areas, particularly in Asia and Africa, are as yet unsurveyed. In the Americas alone, Record and Hess[5] report 530 species of gymnosperms (coniferous species) and about 23,000 species of angiosperms, consisting of trees, and woody vines and shrubs. The United States has more than 800 arborescent species, not all of which, however, are of commercial importance. Of these United States species, systematic strength data are available on 164.

What has science learned about wood, and what does the future hold in store?

As with other materials, it can be taken as axiomatic that a thorough understanding of wood and a basic knowledge of its properties are fundamental to intelligent use. Yet from one viewpoint, wood is unique in that, on the one hand, it can be and often is used without much deliberation while, on the other hand, it presents a greater complexity and variety of problems than perhaps any other single engineering material.

THE FOREST AND THE TREE, AS A "WOOD FACTORY"

> "Large streams from little fountains flow,
> Tall oaks from little acorns grow"
> —*Smith.*

WOOD FORMATION

Wood is an organic material—a renewable resource—the product of the living tree; and it is interesting to contemplate that every tree of the forest represents, species by species, an unbroken life line from ages past.

For the production of wood, nature invented chlorophyll, which, actuated by the energy of sunlight, manufactures sugar out of carbon dioxide gas and water. The green leaves, by the process of photosynthesis, break up the CO_2 molecule, giving back oxygen to the air, and combining carbon with water and other minerals conveyed from the root

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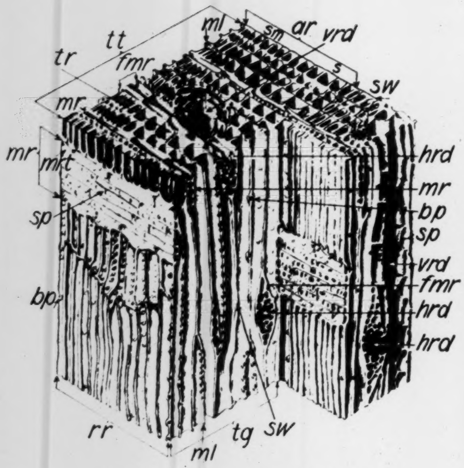
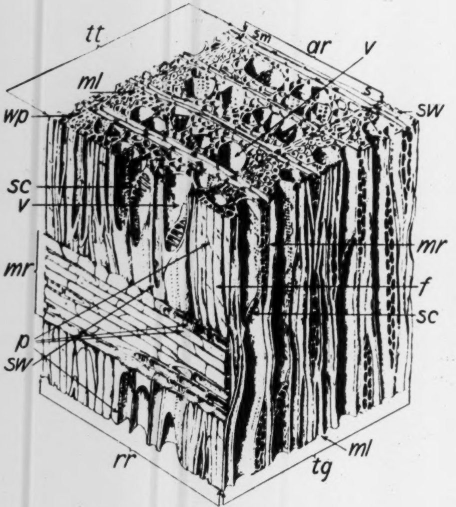
[5] Samuel J. Record and Robert W. Hess, "Timbers of the New World," New Haven, Conn. (1943).



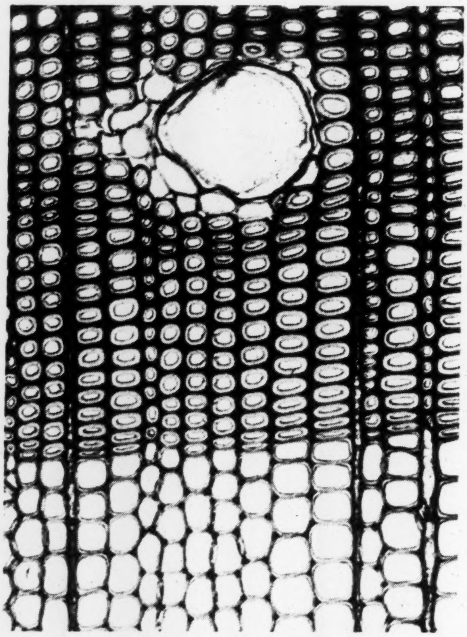
The Structure of a Softwood.

A magnified block of a typical softwood: *ar*, annual growth ring; *bp*, bordered pit; *fmr*, fusiform wood ray; *hrd*, horizontal resin canal or duct; *ml*, middle lamella; *mr*, wood ray; *mrt*, wood ray tracheid; *rr*, radial surface; *s*, springwood; *sm*, summerwood; *sp*, simple pit; *sw*, secondary wall; *tg*, tangential surface; *tr*, tracheid; *tt*, transverse surface; *vrd*, vertical resin canal or duct.



The Structure of a Hardwood.

A magnified block of a typical hardwood: *ar*, annual growth ring; *f*, fiber; *ml*, middle lamella; *mr*, wood ray; *p*, pit; *rr*, radial surface; *s*, springwood; *sc*, scalariform perforation between vessels; *sm*, summerwood; *sw*, secondary wall; *tg*, tangential surface; *tt*, transverse surface; *v*, vessel or pore; *wp*, wood parenchyma.



Cross-Section of Shortleaf Pine.

Showing difference in thickness of fiber walls in springwood and summerwood.

system to form sugar, starch, cellulose, and other food products. A white pine with a trunk diameter of 16 in. may display as many as 6½ million needles, with a surface area of approximately 10,000 sq. ft., which may be 14 to 17 times the ground area covered by the tree.[6] This is the complicated wood "factory," automatically operated by nature. Thus is the energy of the sun converted into wood—and to coal, oil, and gas—which on combustion give up this energy again.

Cell Formation:

A tree grows by the formation of new cells—myriads of new cells—under the bark and at the tips of the branches and roots. To provide food for this

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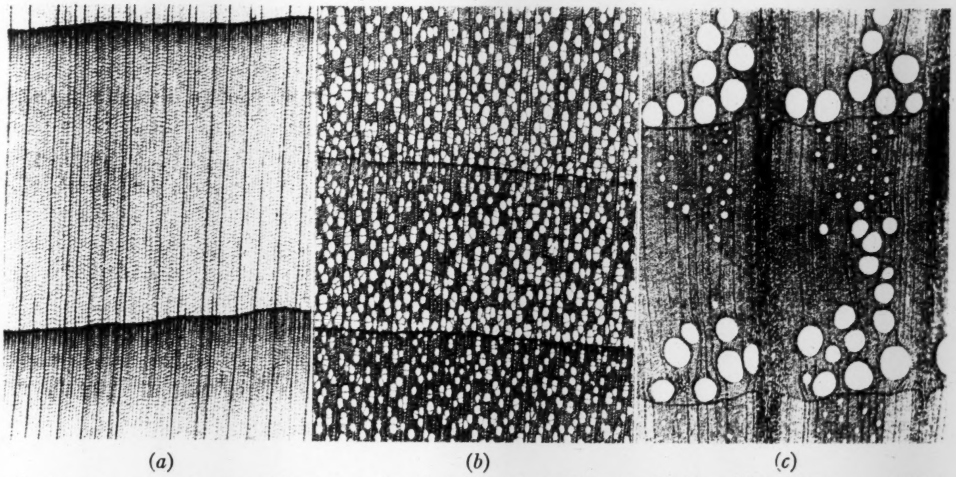
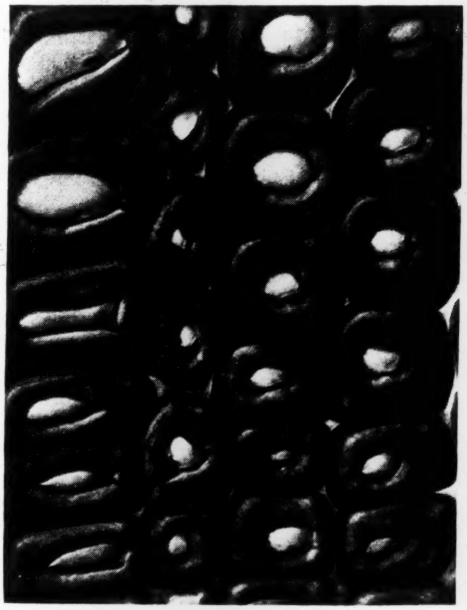
[6] Hans Burger, "Holz, Blattmenge, und Zuwachs, I: die Weymouthsföhre," *Mitteilungen der Eidgen. Anstalt für das Forstliche Versuchswesen*, Vol. 15, Part 2, Zurich (1929).



(*a*) (*b*) (*c*)

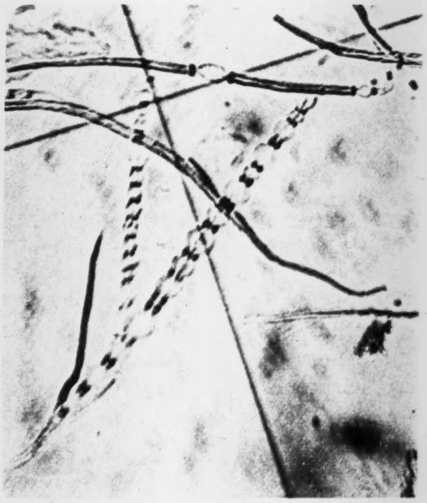
The Structure of Wood as Viewed on the End of a Piece Presents Distinguishing Features Which Make Identification Possible.

These highly magnified sections represent: (*a*) nonporous structure, as represented by coniferous species, such as white pine; (*b*) diffuse-porous structure, with scattered pores of fairly uniform size, as found in certain hardwood species, such as yellow poplar, maple, birch, and beech; and (*c*) ring-porous structure, as found in certain hardwood species such as oak, ash, and elm.



Highly Magnified Cross-Section of Wood Fibers.

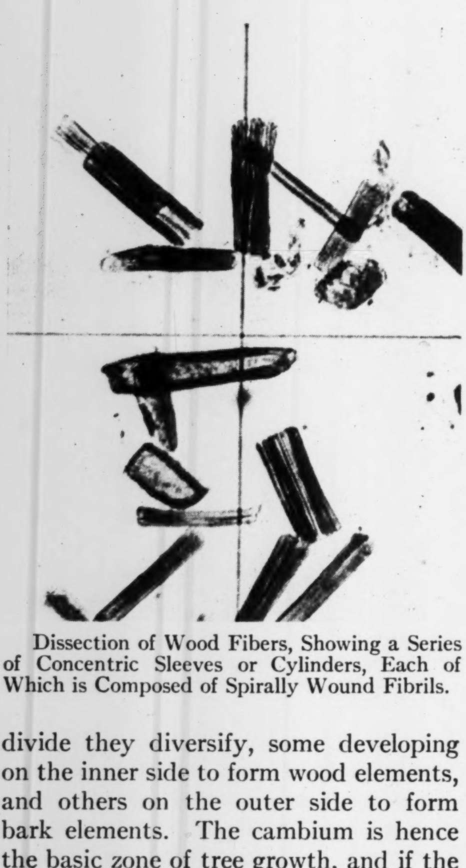
The lignin, which cements the fibers together, has been chemically removed, leaving the hollow fibers appearing as separate units. The chemical pulping of wood in the papermaking industry is essentially a process of dissolving the lignin to separate the fibers.



Highly Magnified Wood Fibers Partially Dissected by Chemical Means.

Removal of parts of the outer wrappings has allowed inner striations to bulge out in the bead-like prominences. These wood fibers are approximately $\frac{1}{7}$ in. long and the thickness is about $\frac{1}{100}$ of the length.

growth, soil moisture with mineral matter in solution moves upward, as sap, through the sapwood to the leaves, while the new compounds of photosynthesis flow downward to the growing parts through the inner bark, and presumably also through the sapwood. The growing region just under the bark is known as the cambium. As the cambium cells divide they diversify, some developing on the inner side to form wood elements, and others on the outer side to form bark elements. The cambium is hence the basic zone of tree growth, and if the cambium is severed by girdling, the tree will soon die. Tree growth year by year hence consists of adding a new and complete layer or envelope over the entire tree structure, the thickness of the growth layer depending on the growth conditions, the species, and many other factors.



Dissection of Wood Fibers, Showing a Series of Concentric Sleeves or Cylinders, Each of Which is Composed of Spirally Wound Fibrils.

Most of the cells are oriented vertically in the tree, and because they are longer than they are wide, they are called fibers.

The fibers as a rule comprise the major part of the wood. In the hardwoods the fibers serve simply as structural elements in the tree, but in the softwoods they function also in the conduction of sap. The softwood fibers, thus serving a dual function, are technically known as tracheids.

But there are also structural differences as well as functional differences between hardwoods and softwoods. Scattered among the fibers of hardwoods are large open-ended cells which form continuous longitudinal passages known as *pores* or *vessels*, which serve to conduct the sap. In certain hardwoods, such as white oak, the pores in the heartwood and inner sapwood become filled with ingrowths called tyloses, which close them from intercommunication. The pores of the red oak, on the other hand, are not filled with tyloses, as a rule, so that air and liquids can readily pass longitudinally through a piece of the wood. According to the size and arrangement of the pores, some of the hardwoods, such as oak, are classified as ring-porous, and others as diffuse-porous. The softwoods are designated as nonporous because the only longitudinal passages present, aside from the tracheids, are resin ducts. There are also horizontal resin ducts.

Scattered among the wood fibers are short, thin-walled cells called wood parenchyma, which are hard to distinguish individually with a hand lens. Collectively, they may appear as bands. While abundant in the hardwoods, wood parenchyma cells are comparatively scarce in the softwoods.

In all woods (except palms, yuccas, and bamboos) there are strips of radial cells, oriented horizontally, known as medullary rays, or rays, the purpose of which is to store food and conduct sap transversely in the tree. The rays are particularly large and conspicuous in some species, such as oak.

These features of structure are important not only because they serve as a means of identification but also because they have an even more significant influence on the application and utility of the various species.

Distinction Between Hardwoods and Softwoods:

The terms "hardwood" and "softwood" are often confusing to those not familiar with the lumber industry. Instead of indicating the hardness or softness of wood, these terms are simply popular descriptive names in use for two great groups botanically known as angiosperms and gymnosperms, but more popularly as "trees with broad leaves" and as "conifers." Although the terms "hardwoods" and "softwoods" are the most generally accepted popular names for the two classes of trees, they are the most misleading. Oak, birch, and basswood are common "hardwood" species, while longleaf pine, spruce, and cypress are "softwoods." While it is true that many "hardwoods," such as oak, are really hard, others, such as basswood, are softer than many "softwoods" (conifers); in fact, balsa, one of the softest woods in the world, is a broad-leaved species, and falls in the so-called "hardwood" group.

Annual Rings:

In climates where temperatures limit tree growth to the summer months, each year's growth increment is usually readily distinguishable and is called an *annual ring*. In tropical climates, on the other hand, growth is more or less continuous but, nevertheless, concentric demarkations occur as a result of fluctuations in growth activity. Such demarkations should not be confused with the annual rings.

Springwood and Summerwood:

In many woods, cells formed in the spring, when tree growth is most active, are comparatively large and thin-walled, whereas, those formed later in the year are smaller and relatively thick-walled. The former constitute the springwood, and the latter the summerwood. Obviously the thin-walled cells are weaker than the thick-walled cells, and hence summerwood is, in general, denser and stronger than springwood. In woods that exhibit a marked contrast in springwood and summerwood, such as Douglas fir and southern yellow pine, the proportion of summerwood offers a visual basis for estimating density and strength.

Sapwood and Heartwood:

In the tree, certain of the wood elements function as living cells for a time, but eventually become inactive. As the tree increases in age and size, an increasing portion of the cells from the pith outward cease to function other than as mechanical support. This inner, and usually darker, portion of the tree is called the heartwood. The outer layers of growth, which contain the only living elements of the wood, are called sapwood. The sapwood is light in color and varies greatly in thickness among different species, among individual trees of the same species, and even among different portions of a tree.

THE WOOD ITSELF

> "We uster watch the steamers an' the hathis pilin' teak,
> Elephints a-pilin' teak,
> In the sludgy, squdgy creek,
> Where the silence 'ung that 'eavy, you was 'arf afraid to speak!
> On the road to Mandalay—"
>
> —*Kipling.*

A background regarding the structure of wood, its chemical composition, physical characteristics, mechanical properties, and conversion is pertinent to its most efficient use.

IDENTIFICATION

The identification of wood is essential not only as a basis for its proper use, but also in its marketing. The most thorough identification of wood, and sometimes the only positive one, is by means of the botanical features of the tree in the forest. The diverse structural characteristics of the different species of wood, however, together with variations in odor, weight, color, and other features of appearance, usually afford a practical means of identification from samples of the wood alone. Frequently a hand lens is sufficient to bring out the structural differences; at other times a higher magnification is required. Identification is based on keys developed from special studies of individual species and so arranged as to classify the several distinguishing features. Keys by different authors may vary with respect to the method and procedure used.

From the wood alone, however, it is not possible to distinguish all individual species, because many related species are so alike. For example, the several species of southern yellow pine cannot be individually identified; but, on the other hand, their properties are generally similar, so that for many uses they can be considered as a group.

PHYSICAL STRUCTURE

Considering first the physical structure, wood has been shown to be made up of small, hollow fibers. The fibers in softwoods are about $\frac{1}{7}$ in. long and 0.07 in. thick, but those in the hardwoods are, on the average, much shorter. Table I presents data on the fiber length of a number of species. The length of the fibers, however, is not a criterion of the strength of the wood. For example, it may be observed that the fibers of

TABLE I.—AVERAGE LENGTH OF FIBERS FOR A NUMBER OF WOODS GROWN IN THE UNITED STATES.[a]

Species	Length, mm.	
	Average	Standard Deviation
Softwoods:		
Longleaf pine (Pinus palustris)	4.90	0.83
White pine (Pinus strobus)	3.67	0.70
Loblolly pine (Pinus taeda)	4.33	0.91
White spruce (Picea glauca)	3.31	0.57
Red spruce (Picea rubra)	3.06	0.57
Balsam fir (Abies balsamea)	3.41	0.51
Eastern hemlock (Tsuga canadensis)	3.80	0.79
Redwood (Sequoia sempervirens)	6.59	1.17
Douglas fir (Pseudotsuga taxifolia)	3.40	0.70
Baldcypress (Taxodium distichum)	4.72	1.03
Western red cedar (Thuja plicata)	3.09	0.46
Northern white cedar (Thuja Occidentalis)	2.16	0.45
Hardwoods:		
White oak (Quercus alba)	1.39	0.20
Red oak (Quercus borealis)	1.32	0.29
Shagbark hickory (Hicoria ovata)	1.34	0.28
Black walnut (Juglans nigra)	1.21	0.14
Yellow birch (Betula lutea)	1.38	0.17
White elm (Ulmus americana)	1.55	0.20
Sugar maple (Acer saccharum)	0.92	0.13
Yellow poplar (Liriodendron tulipifera)	1.74	0.29
White ash (Fraxinus americana)	1.26	0.17
Blackgum (Nyssa sylvatica)	2.30	0.36
Black willow (Salix nigra)	0.85	0.17
Catalpa (Catalpa speciosa)	0.64	0.11

[a] From Brown and Panshin, "Commercial Timbers of the United States."

shagbark hickory, noted for its high strength and toughness, are much shorter than those of black gum, which is inferior to hickory in its mechanical properties. The bond between fibers, regardless of length, is exceedingly strong, and does not constitute a principal source of weakness, as evidenced by the fact that the fibers of wood tested in tension do not commonly separate from one another but rather tear apart.

Microstructure:

Visible under higher magnifications are small structural units of the fiber. The cell walls are made up of concentric layers—a primary, a secondary, and sometimes a tertiary layer. The primary layer is the modified membrane of the original cell. The secondary layer is adjacent to the inner face of the primary and is considerably thicker than the primary layer. The tertiary layer is adjacent to the inner face of the secondary layer, but is generally difficult to distinguish in normal fibers.

The secondary layer is divided into sleeves, the number of which varies from eight to twelve in wood, and to as high as twenty in cotton. The primary layer of the cell wall and the sleeves of the secondary layer can be dissected into long slender fibrils, arranged spirally, which are the smallest units that become evident through any simple mechanical disintegration.

Microscopically visible substructures of the fibrils comprise dermatosomes, ellipsoids, spindle-shaped fusiforms, and spherical units. The ellipsoidal and spherical particles in all probability do not have these shapes in the cell wall, but rather appear to be so shaped when isolated, due to swelling and optical diffraction effects. These may be isolated by treatment of the fibril under carefully controlled conditions. The spherical units, the smallest component of the cell wall visible with the microscope, are about 0.00001 in. in diameter. It is possible, however, by indirect methods using the ultra centrifuge and the X-ray, to determine the approximate size and arrangement of the submicroscopic units.

The largest submicroscopic unit of cellulose was believed to be the micelle, but more recently the name crystallite has been generally applied to the micelle on account of its crystal properties. The crystallite is thought to be composed of long cellulose chains which, in turn, are composed of anhydroglucose residues.

The cellulose molecule $(C_6H_{10}O_5)_x$ has a length many hundreds of times its diameter. It is exceedingly strong in the longitudinal direction, but adjacent molecules are not strongly united crosswise. The cellulose molecules are arranged end to end, and in groups of perhaps 40 to 60 are regarded as constituting a cellulose crystal or micelle. According to the latest evidence, the crystallites or micelles are not separate structural units, but zones of preferred orientation in a thread-like mass of cellulose molecules, part of which extend from one crystallite into another.

CHEMICAL COMPOSITION

Wood fibers, like those of cotton, are composed principally of cellulose and are cemented together with a baffling substance called lignin. Roughly, cellulose comprises about 60 per cent of wood substance; lignin, 28 per cent; and sugars and extractives, the remaining 12 per cent. If wood chips are subjected to a chemical process, the lignin cementing material can be dissolved to free the cellulose fibers. The fibers then become known as pulp, which, after further processing, is made into diversified paper and paperboard products or converted to rayon and other cellulose derivatives.

Holocellulose is the cellulose representing the carbohydrate fraction in extractive-free wood. Results of chemical analyses indicating the holocellulose and lignin content of a number of species of wood are presented in Table II.

Results of complete chemical analyses of a number of widely different species of wood are presented in Table III.

It seems axiomatic that the mechanical properties of wood are dependent on physical structure and chemical composition, yet the chemistry of wood is so complex that no very definite relations have so far been established between mechanical properties or utility and chemical composition.

TABLE II.—RESULTS OF CHEMICAL ANALYSES OF WOOD TO DETERMINE THE HOLOCELLULOSE AND LIGNIN CONTENT.

Based on oven-dry (103 C.) weight of the extractive-free wood.

Wood	Holocellulose by New Method, per cent[a]	Lignin by Sulfuric Acid Method, per cent	Lignin and Holocellulose (Column 1 + Column 2), per cent
White spruce......	73.3	26.6	99.9
Red spruce........	72.9	26.6	99.5
Jack pine.........	72.5	27.2	99.7
Balsam fir........	69.9	30.1	100.0
Eastern hemlock..	68.5	31.5	100.0
Aspen.............	82.5	17.3	99.8
White oak........	75.4	24.1	99.5
Maple............	76.3	23.5	99.8
Willow...........	78.3	22.0	100.3

[a] Method developed by W. G. VanBeckum and G. J. Ritter at the Forest Products Laboratory, Madison, Wis.

Lignin Utilization:

Ever since the process of making pulp was discovered, lignin has been considered the ugly duckling among wood components. Unlike the silky cellulose fibers, it has no definite physical structure. While cellulose has yielded many products now commonplace, like rayon, photographic film, and lacquers, lignin has resisted most efforts to learn more of its chemical makeup and, therefore, has been considered a waste material. A million tons are dumped annually into our streams as waste pulping liquor, causing stream pollution. More than 15 million tons are contained in the waste wood in logging and sawmill operations. The opportunities presented here for waste utilization are challenging.

Attempts to apply the disciplines of chemical utilization to lignin indicated that it is not fully saturated and, therefore, under proper conditions, hydrogen might be added. Following up this lead, lignin was placed in a bomb in the presence of hydrogen and subjected to a pressure of 4000 psi. at 480 F. for several hours. The result was a discovery so far-reaching that its full import will not be known for years. It was surprising to find that the brown powdery material had been almost magically transformed into a transparent liquid. This transparent liquid has been fractionated into methanol (wood alcohol), and four compounds that are all new to the chemical world. The discovery, together with other developments, gives promise of future uses of lignin leading to the possible establishment of new industries.

MOISTURE CONTENT

All the functional development of the tree takes place in the presence of moisture, and all the wood remains "green" or moist throughout the life of the tree. The moist condition is hence the normal one for wood, while the dry condition is abnormal. On the other hand, most uses of wood involve the dry condition, so that drying or seasoning is an important consideration.

The moisture content of wood plays an important part in all commercial applications. Moisture content is the weight of water contained in the wood, expressed as a percentage of the weight of the oven-dry wood. Moisture content is commonly determined by weighing a sample and then drying it at 212 F.

(100 C.) until the weight becomes constant. The loss of weight divided by the weight of the oven-dry wood is the proportion of moisture in the piece. "Moisture" as thus determined is subject to some inaccuracy, because the loss in weight includes that of any substances other than moisture that evaporate at 100 C., but such errors are usually not sufficient to affect the practical application.

The oven-drying method of determining moisture content usually requires one or more days. There have been developed and are now available a number of electrical instruments for the instantaneous determination of moisture content, which work on the principle of electrical resistance or of capacity. Such instruments have certain limitations of range and require calibration, but are nevertheless exceedingly useful.

TABLE III.—RESULTS OF COMPLETE CHEMICAL ANALYSES OF SEVERAL SPECIES OF WOOD.
Results in percentage of oven-dry (105 C.) samples.

Species	Sample	Moisture	Ash	Solubility				Acetic Acid	Methoxy	Pentosan	Methyl pentosan	Cellulose	Lignin	In Cellulose				
				In cold water	In hot water	In ether	In 1 per cent NaOH							Pentosan	Methyl Pentosan	Alpha Cellulose	Beta Cellulose	Gamma Cellulose
Ponderosa pine (Pinus ponderosa)	141...	5.32	0.43	5.58	5.67	9.63	22.08	0.92	4.55	8.06	1.68	56.22	26.75	9.50	2.13	69.18	.....	30.82
	142...	8.28	0.42	2.97	3.40	6.52	16.58	1.24	4.02	9.96	1.24	57.72	25.85	8.97	1.97	66.17	5.52	28.31
	143...	6.89	0.55	4.62	6.33	9.45	23.16	1.03	4.51	5.88	1.81	58.88	26.29	4.20	1.90	57.65	6.54	35.80
	144...	5.19	0.44	3.17	4.89	6.48	19.37	1.18	4.87	5.52	1.77	56.82	27.72	4.63	1.90	55.40	19.02	25.58
	Mean....	6.42	0.46	4.09	5.05	8.52	20.30	1.09	4.49	7.35	1.62	57.41	26.65	6.82	1.98	62.10	10.56	30.13
Alaska yellow-cedar (Chamaecyparis nootkatensis)	161...	3.54	0.33	1.66	2.89	2.67	13.80	1.66	5.42	8.86	3.97	51.45	33.21	8.60	2.03	62.88	11.10	26.02
	162...	5.19	0.35	3.03	3.46	3.34	14.49	1.62	5.60	8.52	3.57	54.04	31.27	8.09	1.91	62.96	10.99	26.05
	163...	4.87	0.62	2.47	2.86	2.08	12.69	1.55	5.05	6.72	2.97	54.78	30.43	6.65	1.65	59.37	11.64	28.97
	164...	5.91	....	2.74	3.23	2.11	12.67	1.54	4.92	7.37	3.17	55.17	30.27	5.86	1.54	65.52	10.51	23.97
	Mean....	4.89	0.43	2.47	3.11	2.55	13.41	1.59	5.25	7.87	3.42	53.86	31.32	7.30	1.78	62.68	11.06	26.25
Incense cedar (Libocedrus decurrens)	165...	5.32	0.38	3.09	4.87	3.33	17.37	0.96	6.34	11.07	1.20	39.94	38.14	9.53	2.13	48.47	15.42	36.11
	166...	4.63	0.27	2.53	3.62	4.69	13.81	1.04	6.29	9.99	1.60	44.10	37.73	7.90	1.71	41.42	12.77	45.81
	167...	5.42	0.38	5.31	7.64	4.90	21.89	0.73	6.09	10.70	1.26	40.76	37.17	9.83	2.13	50.90	6.82	42.28
	Mean....	5.12	0.34	3.64	5.38	4.31	17.69	0.91	6.24	10.65	1.35	41.60	37.68	9.08	1.99	46.92	11.67	41.06
Redwood (heartwood) (Sequoia sempervirens)	168...	9.64	0.22	7.31	9.77	1.00	20.06	1.03	5.27	7.93	2.77	48.67	34.18	7.40	2.09	78.81	2.95	18.24
	168...	9.71	0.20	7.40	9.94	1.14	19.94	1.13	5.16	7.67	2.74	48.23	34.25	.....	....	......	......	.....
	Mean....	9.68	0.21	7.36	9.86	1.07	20.00	1.08	5.21	7.80	2.75	48.45	34.21	7.40	2.09	78.81	2.95	18.24
Tanbark oak (Quercus densiflora)	151...	4.10	0.78	4.14	5.25	0.74	22.59	5.70	5.34	20.02	....	59.40	23.29	23.22	....	55.50	18.81	25.64
	152...	3.95	0.81	4.32	6.15	0.73	25.33	5.70	6.19	20.00	....	56.50	26.07	25.46	....	55.91	2.95	41.14
	153...	3.26	0.82	4.22	5.92	0.80	23.91	4.40	5.35	19.13	....	57.27	25.20	20.30	....	58.15	26.62	19.23
	154...	3.36	0.91	3.72	5.08	0.98	22.93	5.13	6.09	19.22	....	58.95	24.86	22.32	....	57.82	18.27	24.21
	Mean....	3.66	0.83	4.10	5.60	0.80	23.96	5.23	5.74	19.59	....	58.03	24.85	22.82	....	56.77	19.92	23.03
Mesquite (Prosopis juliflora)	171...	4.53	0.57	12.55	15.27	2.20	28.72	1.53	5.68	14.04	0.59	44.79	30.27	17.99	1.07	76.00	1.73	22.27
	172...	6.10	0.58	13.50	15.77	....	30.08	1.70	5.69	13.99	0.69	45.28	30.13	17.99	1.21	76.71	2.56	20.73
	173...	6.15	0.49	12.68	15.56	2.33	29.60	2.53	5.29	13.85	1.18	45.87	31.28	17.57	....	76.40	2.06	21.54
	174...	5.20	0.50	11.74	13.77	2.37	25.69	2.37	5.66	13.95	0.34	45.97	30.22	17.44	0.96	76.83	3.04	20.13
	Mean....	5.49	0.54	12.62	15.09	2.30	28.52	2.03	5.55	13.96	0.70	45.48	30.47	17.75	0.81	76.48	2.35	21.17
Balsa (Ochroma lagopus)	175...	6.50	2.15	1.85	2.84	1.27	20.37	5.75	5.71	17.51	0.88	54.04	26.52	19.99	1.35	75.64	0.27	24.08
	175...	6.44	2.09	1.68	2.74	1.19	20.36	5.85	5.65	17.79	0.83	54.24	26.47	.....	....	......	.....	.....
	Mean....	6.47	2.12	1.77	2.79	1.23	20.37	5.80	5.68	17.65	0.86	54.15	26.50	19.99	1.35	75.64	0.27	24.08
Hickory (shellbark) (Hicoria ovata)	177...	9.60	0.65	4.71	5.41	0.65	18.65	2.60	5.61	18.58	0.92	55.60	23.83	21.33	1.64	75.27	2.64	22.09
	178...	7.39	0.74	4.86	5.73	0.62	19.44	2.42	5.65	19.06	0.67	56.85	23.04	22.45	1.19	77.38	3.01	18.61
	Mean....	8.49	0.69	4.78	5.57	0.63	19.04	2.51	5.63	18.82	0.80	56.22	23.44	21.89	1.41	76.32	2.82	20.35

Moisture in the Tree:

The amount of moisture in the living tree varies widely among species, individual trees of the same species, different parts of the same tree, and between heartwood and sapwood.

Many coniferous species have a large proportion of moisture in the sapwood and much less in the heartwood. Most hardwoods, on the other hand, show more nearly the same moisture content in heartwood and sapwood. Extreme limits observed in the moisture content of green wood range from as low as 30 to 40 per cent in the heartwood for such species as black locust, white ash, Douglas fir, southern pines, and various cedars to about 200 per cent in the sapwood of some coniferous species. In the heartwood of some species the moisture content is high at the base of the tree and becomes less toward the top. For example, in a number of green redwood trees examined, the heartwood decreased in average moisture content from 160 per cent at the stump to 60 per cent at heights above 100 ft. In this instance, however, the moisture content of the sapwood increased slightly with height in tree.

Fiber-Saturation Point:

Moisture in wood in the green state is in part held absorbed by the cell walls and in part within the cell cavities, much in the same manner as water is held in a container. As wood dries, the cell walls do not give off moisture until the adjacent cavities are empty. The condition in which the cell walls are fully saturated and the cell cavities empty is known as the "fiber-saturation point." It varies from 25 to 35 per cent moisture content.

Moisture Content as Related to Strength:

Increase in strength begins when the cell walls begin to lose moisture; that is, after the wood is dried to below the fiber-saturation point. From this point on, most strength properties increase rapidly as drying progresses. This increased strength of dry over green wood of the same dimensions is due to two causes: (1) Actual strengthening and stiffening of the cell walls as they dry out, and (2) increase in the compactness or the amount of wood substance in a given volume because of the shrinkage that accompanies drying below the fiber-saturation point.

TABLE IV.—AVERAGE INCREASE (OR DECREASE) IN VALUE EFFECTED BY LOWERING (OR RAISING) THE MOISTURE CONTENT 1 PER CENT.[a]

	Per Cent
Static bending:	
Fiber stress at proportional limit	5
Modulus of rupture, or cross-breaking strength	4
Modulus of elasticity, or stiffness	2
Work to proportional limit	8
Work to maximum load or shock-resisting ability	0.5
Impact bending:	
Fiber stress at proportional limit	3
Work to proportional limit	4
Height of drop of hammer causing complete failure	0.5
Compression parallel to grain:	
Fiber stress at proportional limit	5
Maximum crushing strength	6
Compression perpendicular to grain:	
Fiber stress at proportional limit	5.5
Hardness, end grain	4
Hardness, side grain	2.5
Shearing strength parallel to grain	3
Tension perpendicular to grain	1.5

[a] The change in strength with change in moisture content occurs only at moisture contents below the fiber-saturation point.

Drying wood to 5 per cent moisture content may add from about $2\frac{1}{2}$ to 20 per cent to its density, as a result of shrinkage, while in small pieces its end-crushing strength and bending strength may easily be doubled and, in some woods, tripled. Thus the first of the two factors mentioned is the one chiefly responsible for the increase in strength.

The increase in strength with seasoning is much greater in small, clear specimens of wood than in large timbers containing defects. In the latter, the increase in strength is to a large extent offset by the influence of defects that develop in seasoning.

The various strength properties are not equally affected by changes in moisture content. Whereas some properties, such as crushing strength and bending strength, increase greatly with decrease in moisture content, others, such as stiffness, change only moderately, and still others, such as shock resistance, may even show a slight decrease. This last effect is due to the fact that drier wood does not bend so far as green wood before failure, although it will sustain a greater load, and because shock resistance or toughness is dependent upon both strength and pliability.

Table IV shows the average variation of the strength properties of wood with change in moisture content, and Fig. 1 shows graphically the effect of moisture on certain strength properties of Sitka spruce.

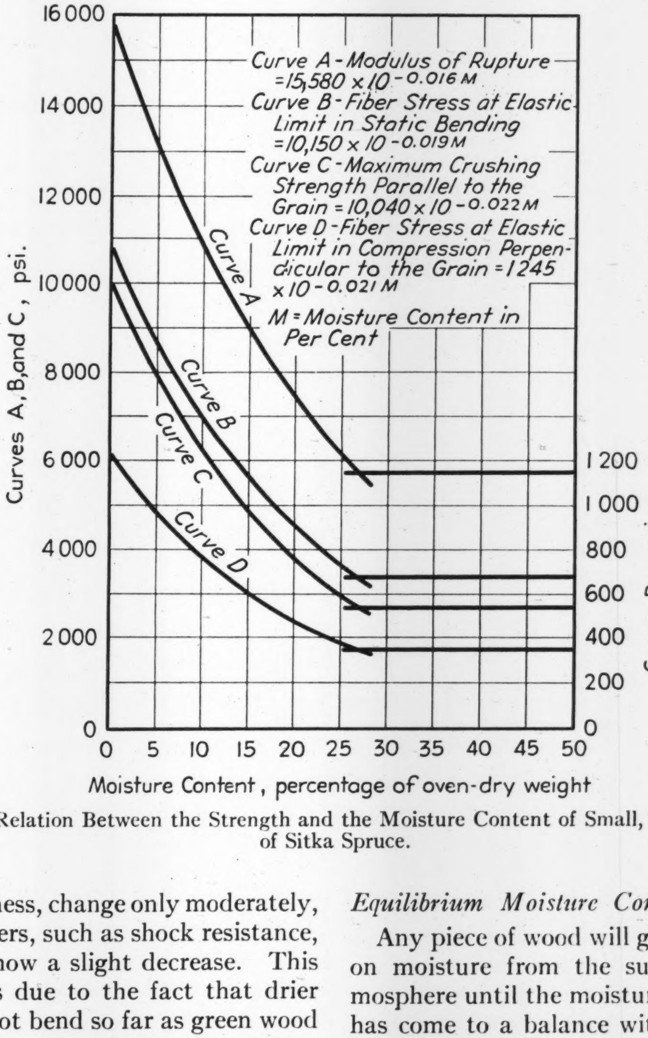


FIG. 1.—The Relation Between the Strength and the Moisture Content of Small, Clear Specimens of Sitka Spruce.

Equilibrium Moisture Content:

Any piece of wood will give off or take on moisture from the surrounding atmosphere until the moisture in the wood has come to a balance with that in the atmosphere. The moisture in the wood at the point of balance is called the equilibrium moisture content. It is a fundamental relationship that the actual amount of moisture in any piece of wood at "equilibrium" is proportional to the amount of wood substance, as indicated by the oven-dry weight of the piece. The actual amount in a dense piece of wood at equilibrium is hence much greater than that in a low density piece of the same volume. Because the percentage of moisture, based on oven-dry weight, correlates with the strength, amount of shrinking and swelling, and other factors, a simple medium of dealing with moisture effects is established, independent of species and variations in density within a species.

Assuming constant temperature, the ultimate moisture content that a given piece of wood will attain, expressed as a percentage of the oven-dry weight, depends entirely upon the relative humidity of the atmosphere surrounding it, which is the amount of vapor in the air expressed as a percentage of the amount it would hold at saturation. This relationship is illustrated by Fig. 2 which shows, for example, that wood kept in an atmosphere constantly at 70 F. and 60 per cent relative humidity will eventually come to a moisture content of about 11 per cent.

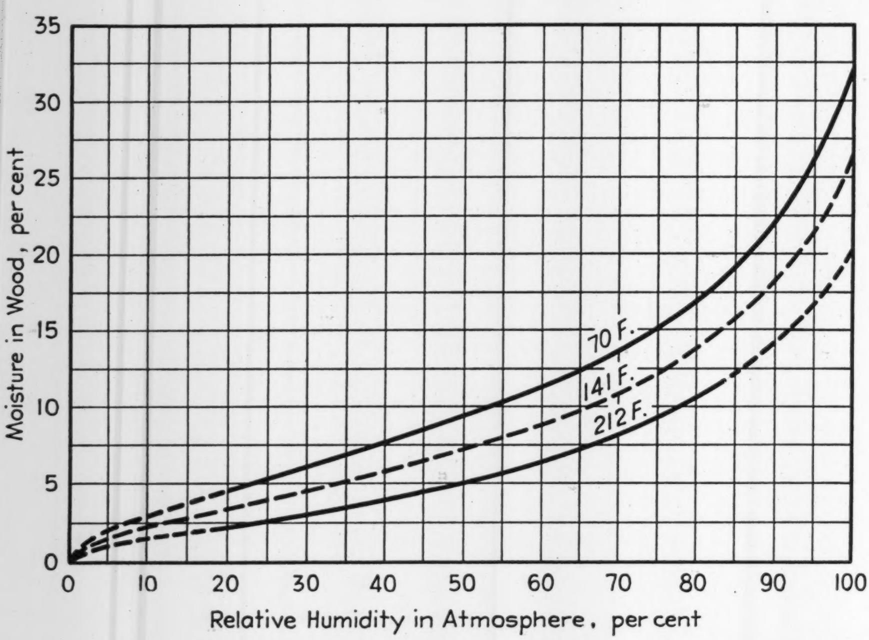


FIG. 2.—Relation of the Equilibrium Moisture Content of Wood to the Relative Humidity of the Surrounding Atmosphere at Three Temperatures.

Shrinkage:

Shrinkage across the grain (in width and thickness) results when wood loses some of the absorbed moisture. Conversely, swelling occurs when dry or partially dry wood is soaked or when it takes moisture from the air or other source. Shrinkage and swelling in the direction of the grain (length) of normal wood is only a small fraction of 1 per cent and is too small to be of practical importance in most uses of wood.

Quarter-sawed (edge-grained) boards shrink less in width but more in thickness than do flat-sawed boards. The greater the difference between radial and tangential shrinkage for a species, the greater is the advantage to be gained through minimizing shrinkage in width by using quarter-sawed wood. The less the difference between radial and tangential shrinkage, the less tendency has wood to check in drying and to cup or warp when its moisture content changes.

The amount of shrinkage in drying is proportional to the moisture lost below the fiber-saturation point. Approximately one half the total shrinkage possible has occurred in wood seasoned to an air-dry condition (12 to 15 per cent moisture content) and about three fourths in lumber kiln-dried to a moisture content of about 7 per cent. The most satisfactory performance of wood, with respect to stabilization of dimension changes, is attained by using properly seasoned and manufactured stock, installed at a moisture content in accord with its service conditions.

In general, the heavier species of wood shrink more across the grain than the lighter ones. Heavier pieces also shrink more than lighter pieces of the same species.

CHEMICAL SEASONING

The living tree contains great quantities of water that must be removed before the lumber into which it is cut is ready for building a home or for many of its other uses. The trunk of a large redwood tree 5 ft. in diameter and 200 ft. long contains more than 17 tons of water, more water than wood by weight, and enough to fill a modest backyard swimming pool. Improved kilns for rapid seasoning of lumber have been developed, but in all the common seasoning methods, such as air drying and kiln drying, the wood dries from the outside in, and care must be taken to avoid serious checking, which is the principal cause of seasoning losses. Newest of the drying methods, known as chemical seasoning, reverses the process by causing the wood to dry from the interior to the exterior. The green wood may either be soaked in a chemical solution or dry chemical may be applied to its surface. In either instance, the wood is in contact with the chemical for a sufficient length of time to permit diffusion of the chemical into the outer zone of the wood surface. The chemical solution lowers the vapor pressure at the surface, while the normal vapor pressure of the moisture at the center of the piece is maintained. Thus moisture moves from the interior to the surface of the timber in response to this vapor pressure gradient, without the necessity of the normal moisture gradient.

While still in its infancy, this method has already won wide acclaim. It is used in many mills in the Pacific Northwest and recently has made possible the rapid seasoning and delivery of millions of feet of timbers for war uses. Seasoning losses in some of the large timbers, which formerly ran from 40 to 60 per cent, have been reduced to less than 5 per cent. Not only are seasoning losses reduced, but the drying time is also decreased.

Douglas fir timbers, 6 by 12 in. in cross-section, that require a year or more to air season can be satisfactorily chemically seasoned with urea in 34 days. Of this elapsed time, 8 days are required for the chemical treatment and 26 days for the kiln.

Comparative Strength of Air-Dried and Kiln-Dried Wood:

Some wood users contend that kiln-dried wood is brash and not equal in strength to wood that is air dried. Others advance figures purporting to show that kiln-dried wood is much stronger than air dried. Extensive strength tests on kiln-dried and air-dried specimens of 28 common species of wood show, however, that good kiln drying and good air drying have the same effect upon the strength of wood, but that severe conditions in the kiln will lower most of the strength properties.

The belief that kiln drying produces stronger wood than air drying is usually the result of failure to consider differences in moisture content. The moisture content of wood on leaving the kiln is generally from 2 to 6 per cent lower than that of thoroughly air-dried stock. Since wood rapidly increases in most strength properties with loss of moisture, higher strength values may be obtained from kiln-dried than from air-dried wood. Such a difference in strength is not permanent, since in use a piece of wood will come to practically the same moisture condition whether it is kiln dried or air dried.

It must be emphasized that the appearance of wood is not a reliable criterion of the effect the drying process may have upon its strength. The strength properties may be seriously injured without visible damage to the wood. Also, it has been found that the same kiln-drying process cannot be applied with equal success to all species. To insure kiln-dried wood of the highest strength, it is essential to follow the correct kiln schedules, such as have already been established for the species, grade, and thickness of material.

ELECTRICAL AND THERMAL PROPERTIES

Electrical Resistance of Wood:

The electrical resistance of wood varies greatly with change in moisture content, especially below the fiber-saturation point, decreasing as the moisture content increases. It also varies slightly with species, is greater across the grain than along it, and approximately doubles for each drop in temperature of 22.5 F. Wood is about as good an electrical insulator as exists when it is oven dry (specific resistance 10^8 ohms per cu. cm.). At the fiber-saturation point its specific resistance is about the same as that of water (10^5 to 10^6 ohms per cu. cm.).

Thermal Expansion of Wood:

Most substances expand more or less when heated. For most wood applications, the thermal expansion is unimportant. Different investigators are not in close agreement in their values for this property of wood, although they do agree that the expansion across the grain is much greater than that along the grain for species on which data are available. Values of the coefficient of thermal expansion of wood per degree Fahrenheit at ordinary temperatures range from 0.0000011 for yellow birch to 0.0000036 for black walnut parallel to the direction of the fibers; and from 0.0000146 for yellow birch to 0.0000341 for American beech across the grain.

Thermal Conductivity of Wood:

The thermal conductivity of wood varies with species, the heavier kinds having higher conductivity values. Since thermal conductivity is inversely proportional to heat-insulating value, the lighter species are the better insulators. Thermal-conductivity values are given in Table V rather than heat-insulating values, because of the comparative ease of determination.

DENSITY AND ITS RELATION TO PROPERTIES

Density of Wood Substance:

Individual cells vary greatly in their size and wall thickness, and it is common knowledge that different species exhibit striking differences in the density of the dry wood. Yet it is significant that the density of wood substance itself is practically constant for all species, being about 1.53. Wood substance, therefore, is heavier than water, and corresponds closely to cotton, which has a specific gravity of 1.59.

With this fact in mind, it is obvious that the only reason some woods float is because of the vacant spaces they contain. A lightweight wood, such as balsa, which has a density of 0.10 to 0.25 is composed of very thin-walled cells with large air spaces; whereas the very dense woods, such as black ironwood, greenheart, and lignum-vitae, with densities of 1.0 to 1.3, are composed of thick-walled cells with relatively small voids. These examples represent the ranges from extremely buoyant woods to those that will sink even when thoroughly dry.

It is of interest to note also that the densest and lightest woods are native to tropical or semi-tropical countries. In the species native to climates where freezing occurs, the thickness of the cell wall and structure is such that the wood is not injured by the freezing of the sap or moisture in the tree.

TABLE V.—THERMAL CONDUCTIVITY ACROSS THE GRAIN OF DIFFERENT WOODS AT MOISTURE CONTENT INDICATED.

Species	Moisture Content, per cent	Mean Temperature, T, deg. Fahr.	Weight, D, lb. per cu. ft.	Thermal Conductivity, K*	Authority
Ash, white	12	75	40.0	1.05	F. B. Rowley, University of Minnesota
Balsa		90	7.3	0.33	Bureau of Standards
		90	8.8	0.38	
		90	20.0	0.58	
Birch, yellow	12	75	43.0	1.00	F. B. Rowley, University of Minnesota
Cedar, western red	12	75	25.0	0.72	
Cypress, southern	12	75	29.0	0.83	
Douglas fir	12	75	33.0	0.77	
Elm (soft)	12	75	32.5	0.91	
Fir, white	12	75	25.0	0.65	
Hemlock, eastern	12	75	28.5	0.80	
Hemlock, western	12	75	28.5	0.76	
Larch, western	12	75	39.0	0.99	
Maple (hard)	12	75	44.5	1.16	
Maple (soft)	12	75	39.0	1.04	
Oak, red	12	75	45.0	1.20	
Oak, white	12	75	46.5	1.22	
Pine, longleaf	12	75	38.0	0.96	
Pine, northern white	12	75	27.5	0.83	
Pine, Norway	12	75	30.0	0.84	
Pine, ponderosa	12	75	30.5	0.85	
Pine, shortleaf	12	75	34.0	0.98	
Pine, sugar	12	75	25.5	0.69	
Redwood	12	75	26.0	0.76	
Spruce, Sitka	12	75	26.5	0.68	

* In British thermal units per hour and per square foot of conducting material, with a temperature gradient of 1 F. per inch of thickness.

Variability:

Variability is common to all materials, but materials differ considerably in the amount of variation or the spread of values.

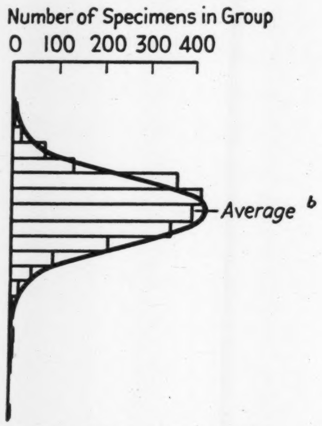
The growing tree is subject to numerous constantly changing influences that affect the wood produced, and it is not surprising that even the clear wood is variable in strength and other properties. The factors affecting tree growth include soil, moisture, temperature, growing space, and heredity.

Everyone who has handled and used lumber has encountered variability and observed that different pieces, even of the same species, are not exactly alike. The differences most commonly recognized are in the appearance, but even greater differences in weight and in strength properties occur and are of greater importance.

The variability of wood can be illustrated by considering the specific gravity frequency distribution of different samples of Sitka spruce as presented in Table VI.

It may be noted that the specific gravity of the heaviest piece included in the series was 2⅔ times that of the lightest, and that the number of very heavy and very light pieces is quite small. Most of the values are grouped quite closely about the average.

TABLE VI.—RESULTS OF SPECIFIC GRAVITY DETERMINATION ON 2105 SAMPLES OF SITKA SPRUCE.

Specific Gravity Group Limits[a]		Pieces in Group		Variability Diagram
Minimum	Maximum	Number	Fraction of Grand Total, per cent	
0.220	0.239	1	0.05	
0.240	0.259	3	0.14	
0.260	0.279	18	0.86	
0.280	0.299	70	3.33	
0.300	0.319	133	6.32	
0.320	0.339	359	17.05	
0.340	0.359	411	19.53	
0.360	0.379	392	18.62	
0.380	0.399	345	16.39	
0.400	0.419	211	10.02	
0.420	0.439	91	4.32	
0.440	0.459	43	2.04	
0.460	0.479	16	0.76	
0.480	0.499	3	0.14	
0.500	0.519	1	0.05	
0.520	0.539	4	0.19	
0.540	0.559	2	0.09	
0.560	0.579	1	0.05	
0.580	0.599	0	0.00	
0.600	0.619	0	0.00	
0.620	0.639	1	0.05	

[a] Based on weight of wood when oven-dry (moisture-free) and volume when green.
[b] Average specific gravity, 0.364; highest observed specific gravity, 0.626; lowest, 0.236.

Assuming a so-called normal distribution and representative material, the calculated probable variation of an individual piece from the average specific gravity is 7.5 per cent. By actual count, 1089 or 51.7 per cent of the pieces studied have a specific gravity between 0.337 and 0.391, whereas 522 or 24.8 per cent were below 0.337 and 494 or 23.5 per cent were above 0.391. As might be expected, the percentages determined by actual count do not agree exactly with the theoretical percentages, but the agreement is sufficiently close to show the value of the theory in estimating the variability even when a normal distribution is assumed. The frequency distribution of the specific gravity values for these 2105 samples of Sitka spruce is shown as a diagram in Table VI.

STRENGTH OF WOOD

Systematic studies have been made at the Forest Products Laboratory on the mechanical properties of woods grown in the United States, and to date, comparable data are available on 164 species. The test methods used in the evaluation of properties are those now described in A.S.T.M. Standard Methods of Testing Small Clear Specimens of Timber (D 143 - 27).[7]

To obtain comparable data on the strength of different species, the material was identified botanically in the forest, and brought to the testing station in log form in an unseasoned condition. Tests were then conducted on specimens in both the green and the seasoned conditions.

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[7] 1942 Book of A.S.T.M. Standards, Part II, p. 616.

Extensive research on the mechanical properties of wood has also been carried on in a number of other countries, particularly England, Canada, Australia, India, and the Union of South Africa. In many instances, the same test procedures have been employed, so that world-wide unification and comparability of results are obtained. Further efforts at standardization have been under way for some years by the Timber Research Committee of the International Union of Forest Research Organizations.

TABLE VII.—COMPARISON OF THE SPECIFIC GRAVITY AND THE MAXIMUM CRUSHING STRENGTH OF MASTIC AND BALSA.

Species	Specific Gravity, Based on Weight and Volume of Wood when Oven-Dry	Maximum Crushing Strength Parallel to Grain, psi.	Specific Strength $\left(\frac{\text{Column 3}}{\text{Column 2}}\right)$
(1)	(2)	(3)	(4)
Mastic......	1.03	5880	5710
Balsa.......	0.11	644	5850

TABLE VIII.—SPECIFIC GRAVITY — STRENGTH RELATIONS AMONG DIFFERENT SPECIES.[a]

Property	Moisture Condition	
	Green	Air-Dry (12 per cent moisture content)
Static bending:		
Fiber stress at proportional limit, psi....................	10 200 $G^{1.25}$	16 700 $G^{1.25}$
Modulus of rupture, psi..................................	17 600 $G^{1.25}$	25 700 $G^{1.25}$
Work to maximum load, in.-lb. per cu. in................	35.6 $G^{1.75}$	32.4 $G^{1.75}$
Total work, in.-lb. per cu. in............................	103 G^2	72.7 G^2
Modulus of elasticity, psi................................	2 360 000 G	2 800 000 G
Impact bending:		
Fiber stress at proportional limit, psi..................	23 700 $G^{1.25}$	31 200 $G^{1.25}$
Modulus of elasticity, psi................................	2 940 000 G	3 380 000 G
Height of drop, in...	114 $G^{1.75}$	94.6 $G^{1.75}$
Compression parallel to grain:		
Fiber stress at proportional limit, psi....................	5 250 G	8 750 G
Maximum crushing strength, psi..........................	6 730 G	12 200 G
Modulus of elasticity, psi.................................	2 910 000 G	3 380 000 G
Compression perpendicular to grain: Fiber stress at proportional limit, psi..................................	3 000 $G^{2.25}$	4 630 $G^{2.25}$
Hardness, lb.:		
End..	3 740 $G^{2.25}$	4 800 $G^{2.25}$
Radial..	3 380 $G^{2.25}$	3 720 $G^{2.25}$
Tangential..	3 460 $G^{2.25}$	3 820 $G^{2.25}$

[a] The values listed in this table are to be read as equations, for example: Modulus of rupture for green material = 17,600 $G^{1.25}$ where G represents the specific gravity, oven-dry, based on volume at moisture condition indicated.

Specific Gravity (or Density) as Related to Strength:

Since the specific gravity of a piece of dry wood is an index of the amount of wood substance it contains, it is also an index of the strength properties. The relations between specific gravity and other properties of wood may be considered on the basis of (1) different pieces of the same species, and (2) different species.

Specific Gravity – Strength Relationship Among Species.—The general relation of specific gravity to strength is illustrated by two widely different woods, mastic, a very heavy species growing in Florida, and balsa, a very light species from Central America. Compression parallel-to-grain tests on green material gave the results in Table VII and show that mastic, with an average specific gravity nine times as great as that of balsa, was nine times as high in crushing strength along the grain. Weight for weight, the crushing strengths parallel to grain of these diverse species are substantially equal.

The average specific gravity – strength relations based on 163 species of hardwoods and softwoods show that some properties, such as maximum crushing strength parallel to grain, increase approximately in proportion to the increase in specific gravity, whereas others increase more rapidly. Modulus of rupture, for instance, varies from one species to another as the $1\frac{1}{4}$ power of specific gravity. Other properties are related to specific gravity by equations of still higher powers; for example, the exponent of specific gravity for relation to hardness is $2\frac{1}{4}$. It is evident, therefore, that small differences in specific gravity may result in large differences in certain strength properties. For example, one species twice as high in specific gravity as another has $4\frac{3}{4}$ times the hardness.

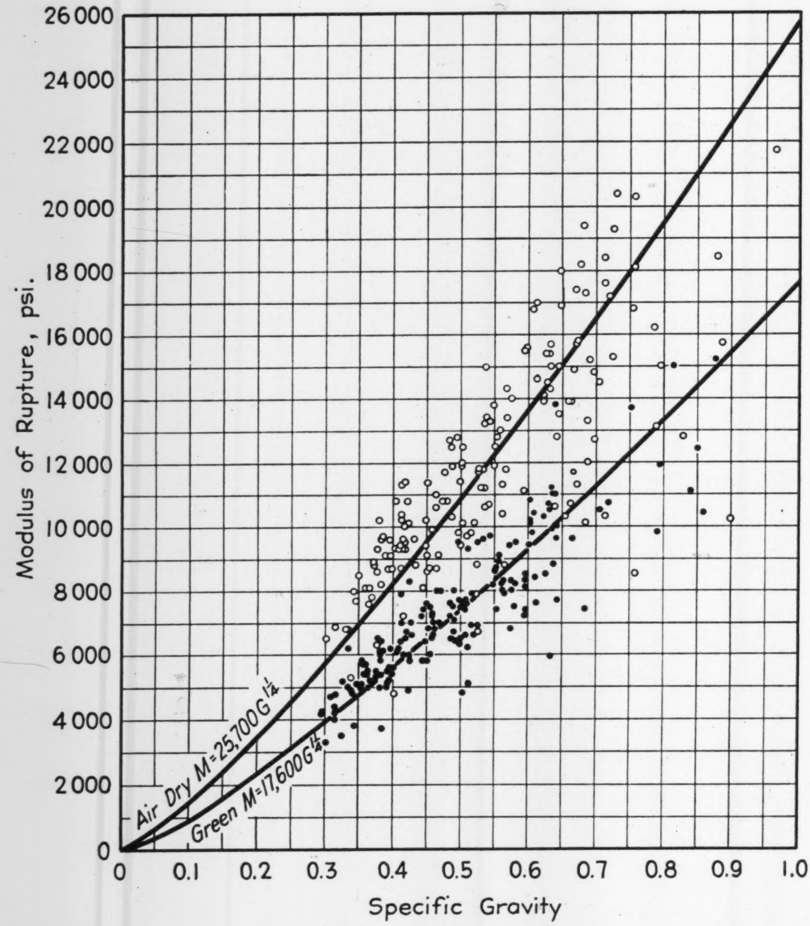


FIG. 3.—Relation of Modulus of Rupture to Specific Gravity for Green and Air-Dry Material of Various Species.

Approximate average relations of specific gravity to strength properties among different species are given in Table VIII.

Some species of wood contain relatively large amounts of resins, gums, and other extractives which add to the weight but do not contribute so much to the strength as would a like amount of wood substance. In addition, species vary in the structural arrangement of their fibers. For these reasons, two species which average the same in specific gravity may exhibit different strength characteristics. This fact is illustrated by the scattering of the points in Fig. 3. Douglas fir (coast type) and sweet gum illustrate an extreme example of variations from the average density-strength relations among species. Although these woods are about equal in weight per unit volume when dry, Douglas fir averages 39 per cent higher in compressive strength but considerably lower than sweet gum in shock resistance.

It is true, likewise, that some species of wood are equal in certain respects to others of higher density. Douglas fir (coast type), although its density is but three fourths that of commercial white oak, is about equal to the oak in bending and compressive strengths, and excels it in stiffness. The oak, however, averages much higher than Douglas fir in hardness and shock resistance. Hence the specific gravity relationships among species represent general trends and not uniform laws. Departure of a species from the general relationship often indicates some exceptional characteristic which makes this species particularly desirable for certain use requirements.

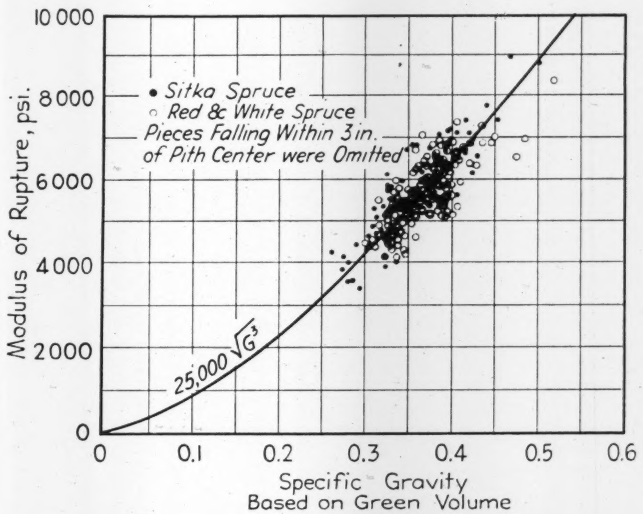


FIG. 4.—Relation of Modulus of Rupture to Specific Gravity. Based on small clear specimens tested in a green condition. Sitka spruce, red, and white spruce.

Specific Gravity – Strength Relationship Among Individual Pieces of a Species.—While a general relationship exists between the specific gravities and strength properties among different species, specific gravity affords a still better index of strength within a species. The heaviest pieces of any species of wood are generally two to three times as high in specific gravity as the lightest ones of the species, and are correspondingly stronger. The relationship among individual pieces of a species is usually represented by a power of specific gravity slightly higher than that representing average values for different species.

Furthermore, departures from the average relationship are less marked. Figure 4 illustrates the relation between the specific gravity and the modulus of rupture for individual pieces of Sitka spruce.

TABLE IX.—AVERAGE STRENGTH AND RELATED PROPERTIES OF SITKA SPRUCE.

Tests made in accordance with A.S.T.M. Standard D 143 - 27, except tension parallel to grain, toughness, Poisson's ratio, and plate shear.

Item	Property	Average Value	
		Green	Dry
1	Moisture content, per cent	42	12
2	Specific gravity[a]	0.37	0.40
3	Weight, lb. per cu. ft.	33	28
4	Shrinkage, volumetric, per cent[b]	11.5	....
5	Shrinkage, radial, per cent[b]	4.3	....
6	Shrinkage, tangential, per cent[b]	7.5	....
	Static Bending:		
7	Fiber stress at proportional limit, psi	3 300	6 700
8	Modulus of rupture, psi	5 700	10 200
9	Modulus of elasticity, psi	1 230 000	1 570 000
10	Work to proportional limit, in.-lb. per cu. in.	0.53	1.62
11	Work to maximum load, in.-lb. per cu. in.	6.3	9.4
12	Total work, in.-lb. per cu. in.	18.3	17.2
	Impact Bending:		
13	Fiber stress at proportional limit, psi	8 400	11 400
14	Work to proportional limit, in.-lb. per cu. in.	3.0	4.2
15	Height of drop causing failure (50-lb. hammer), in.	24	25
	Compression Parallel to Grain, psi.:		
16	Fiber stress at proportional limit	2 240	4 780
17	Maximum crushing strength	2 670	5 610
	Compression Perpendicular to Grain, psi.:		
18	Fiber stress at proportional limit	340	710
	Hardness, lb.:		
19	End	430	760
20	Radial	340	500
21	Tangential	360	520
	Shear Parallel to Grain, psi.:		
22	Radial	740	1 130
23	Tangential	780	1 170
	Cleavage Strength, lb. per in. of width:		
24	Radial	130	180
25	Tangential	160	240
	Tension Perpendicular to Grain, psi.:		
26	Radial	210	310
27	Tangential	290	440
28	Tension Parallel to Grain, psi	8 110	16 960
	Toughness, in.-lb. per cu. in.:		
29	Radial	....	29.2
30	Tangential	....	38.7
	Compression:[c]		
31	Modulus of elasticity (longitudinal), psi	....	1 679 000
32	Modulus of elasticity (radial), psi	....	129 000
33	Modulus of elasticity (tangential), psi	....	76 000
34	Poisson's ratio (longitudinal-radial)[d]	....	0.368
35	Poisson's ratio (longitudinal-tangential)	....	0.464
36	Poisson's ratio (radial-longitudinal)	....	0.039
37	Poisson's ratio (radial-tangential)	....	0.420
38	Poisson's ratio (tangential-longitudinal)	....	0.027
39	Poisson's ratio (tangential-radial)	....	0.255
	Plate Shear, psi.:		
40	Modulus of rigidity (longitudinal-radial)[e]	....	118 000
41	Modulus of rigidity (longitudinal-tangential)	....	112 000
42	Modulus of rigidity (radial-tangential)	....	5 100

[a] Based on oven-dry weight and volume at test.

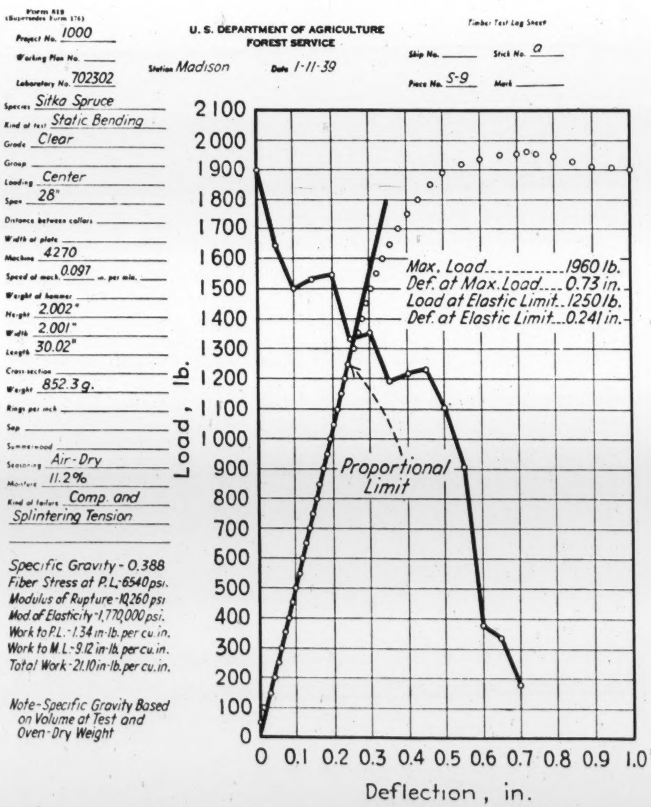
[b] Shrinkage from green to oven-dry condition, based on dimensions when green.

[c] Items, Nos. 31 to 39, inclusive, determined on spruce of average specific gravity of 0.382.

[d] Radial strain divided by longitudinal strain, under longitudinal stress.

[e] Shear from forces acting longitudinally and radially in the longitudinal-radial plane.

Properties of Sitka Spruce.—It may be of interest to consider the properties of a single species, taking Sitka spruce as an example, and examine the variety and relationship of properties, as presented in Table IX. Results are given for both green and dry material. Presented also are typical data sheets



Form 812 (Supersedes Form 176)

U. S. DEPARTMENT OF AGRICULTURE
FOREST SERVICE

Timber Test Log Sheet

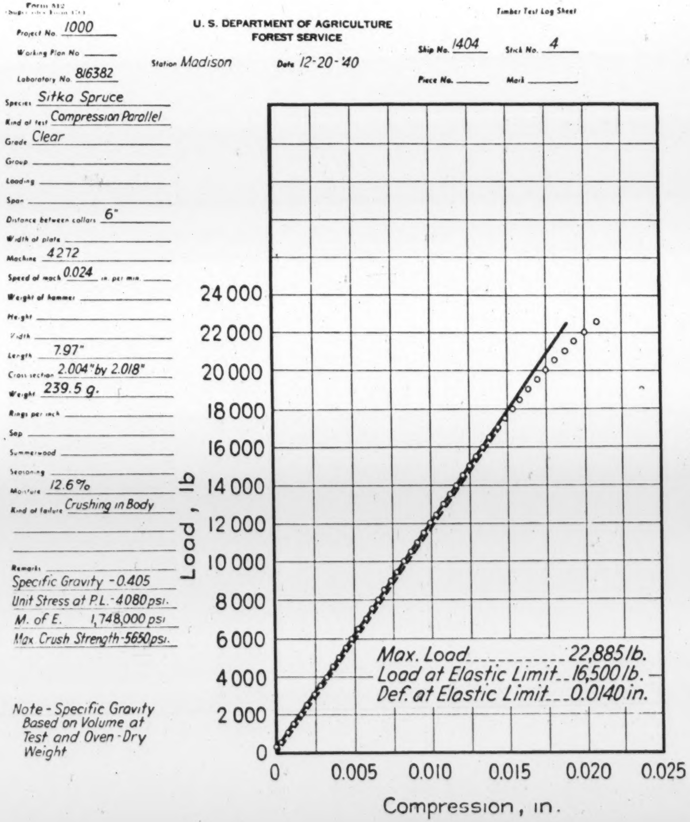
Project No. 1000
Working Plan No.
Laboratory No. 702302
Station Madison
Date 1-11-39
Ship No.
Stick No. a
Piece No. S-9
Mark
Species Sitka Spruce
Kind of test Static Bending
Grade Clear
Group
Loading Center
Span 28"
Distance between collars
Width of plate
Machine 4270
Speed of mach. 0.097 in. per min.
Weight of hammer
Height 2.002"
Width 2.001"
Length 30.02"
Cross-section
Weight 852.3 g.
Rings per inch
Sap
Summerwood
Seasoning Air-Dry
Moisture 11.2%
Kind of failure Comp. and Splintering Tension

Specific Gravity - 0.388
Fiber Stress at P.L.-6540 psi.
Modulus of Rupture-10,260 psi
Mod. of Elasticity-1,770,000 psi.
Work to P.L.-1.34 in-lb. per cu. in.
Work to M.L.-9.12 in-lb. per cu. in.
Total Work-21.10 in-lb. per cu. in.

Note-Specific Gravity Based on Volume at Test and Oven-Dry Weight

Load, lb.
2100
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0
Deflection, in.
Max. Load............1960 lb.
Def. at Max. Load....0.73 in.
Load at Elastic Limit..1250 lb.
Def. at Elastic Limit...0.241 in.
Proportional Limit

FIG. 5.—Typical Load-Deflection Curve Obtained in Standard Static Bending Test of Sitka Spruce.



Form 812

U. S. DEPARTMENT OF AGRICULTURE
FOREST SERVICE

Timber Test Log Sheet

Project No. 1000
Working Plan No
Laboratory No. 816382
Station Madison
Date 12-20-'40
Ship No. 1404
Stick No. 4
Piece No.
Mark
Species Sitka Spruce
Kind of test Compression Parallel
Grade Clear
Group
Loading
Span
Distance between collars 6"
Width of plate
Machine 4272
Speed of mach 0.024 in. per min.
Weight of hammer
Height
Width
Length 7.97"
Cross section 2.004" by 2.018"
Weight 239.5 g.
Rings per inch
Sap
Summerwood
Seasoning
Moisture 12.6%
Kind of failure Crushing in Body

Remarks
Specific Gravity -0.405
Unit Stress at P.L.-4080 psi.
M. of E. 1,748,000 psi
Max Crush Strength-5650 psi.

Note - Specific Gravity Based on Volume at Test and Oven-Dry Weight

Load, lb
24 000
22 000
20 000
18 000
16 000
14 000
12 000
10 000
8 000
6 000
4 000
2 000
0
0 0.005 0.010 0.015 0.020 0.025
Compression, in.
Max. Load............22,885 lb.
Load at Elastic Limit..16,500 lb.
Def. at Elastic Limit...0.0140 in.

FIG. 6.—Typical Load-Compression Curve Obtained in Compression Parallel to Grain Test of Sitka Spruce.

(Figs. 5 and 6) for compression parallel-to-grain and static bending specimens tested in accordance with A.S.T.M. Method D 143.

MECHANICS OF WOOD

> "It should be so built that it *couldn'* break daown
> 'Fur,' said the Deacon, ' 't's mighty plain
> Thut the weakes' place mus' stan' the strain;
> 'n' the way t' fix it, uz I maintain,
> Is only jest
> T' make that place uz strong uz the rest.' "
> —*Holmes.*

AXES OF SYMMETRY

The character, orientation, and arrangements of the fibers make wood an anisotropic material. For all practical purposes, however, it may be treated as orthotropic, with three principal axes of symmetry (Fig. 7), the longitudinal (L), the radial (R), and the tangential (T).

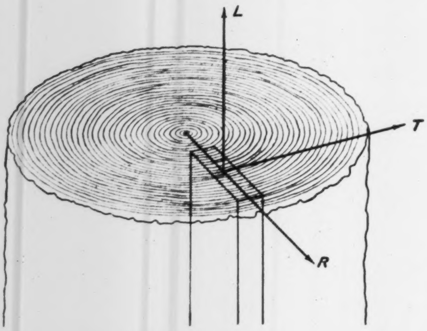


FIG. 7.—Diagrammatic Sketch Showing the Three Principal Axes of Symmetry of Wood.

The assumption of three structural axes results in three Young's moduli varying as much as 150 to 1, three shear moduli varying 20 to 1, six Poisson's ratios varying 40 to 1, and other properties varying with grain direction. These structural axes relationships, employed with proper assumptions, make possible the application of the theory of elasticity in the analysis of wood and plywood elements and structures.

FORM FACTORS

The engineer must depend in his strength calculations on various formulas, many of which are of long standing. The derivation of these formulas is, of course, based on certain assumptions, and the reasonableness of these assumptions in a wide range of application is attested to by their satisfactory use. On the other hand, it is essential that the assumptions upon which the formulas are based, and their limitations, be kept in mind to avoid serious error.

Let us in this connection examine the usual flexure theory:

$$M = S\frac{I}{c} \quad \ldots\ldots\ldots\ldots (1)$$

where:

M = the bending moment,
S = the unit stress in extreme fiber,
I = the moment of inertia, and
c = the distance from neutral axis to outermost fiber.

In this theory it is assumed, among other things, that a plane section remains a plane section, that the stress does not exceed the proportional limit, that the Young's moduli in tension and compression are equal, and that the maximum stress for a material is a constant. In estimating the bending strength of wood, any errors in performance are frequently attributed to errors in the first two of these assumptions. On the contrary, it has been found that the ordinary assumption as to distribution of stress holds quite well up to the proportional limit, and that it is the maximum and proportional limit stresses that vary, being dependent on the size and form of the beam. As long as the results of tests in static bending are applied to members of the same kind, form, size, and condition of loading, the same proportional limit and maximum stress obtains and but little error results even when the stresses exceed the proportional limit: When applied to radically different sections, however, the same proportional limit and maximum stresses *do not* obtain and large errors may result. These errors, in most instances, are away from the side of safety.

Standard strength tests of different species of wood are commonly made on specimens 2 by 2 in. in cross-section. Since, as has been mentioned, test results are, for many properties, intimately related to method, some of these data are consequently strictly applicable only to specimens of the size employed. In the usual flexure formula, the factor by which the proportional limit and maximum stress, S, (based on the results of standard tests) must be multiplied in calculating the strength of any section is known as the form factor of that section. The bending formula is thus simply expressed:

$$M = FS\frac{I}{c} \quad \ldots\ldots\ldots (2)$$

where F represents the form factor.

Form factors for wood have been investigated and established from theoretical and empirical considerations at the Forest Products Laboratory and are applicable to both proportional limit and ultimate stress calculations.

To obtain a better picture of the principles of the form factor theory, a few specific examples will be of interest. Although there is every reason to believe that the ordinary assumption as to distribution of stress in bending holds well to the proportional limit, a wood I beam of a certain size and form, for example, may have an elastic limit stress 30 per cent less than a solid rectangular beam made of the same material. Such an I beam would have a proportional limit form factor, F_E, of 0.70.

Consider, as a further example, a beam of square section with the diagonal vertical. In the ordinary beam formula, Eq. 1, it is seen that the bending moment sustained by such a beam, other things being equal, is directly proportional to I, the moment of inertia, and inversely proportional to c, the distance from the neutral axis to the extreme fiber in compression. It happens that the moment of inertia of a square about a neutral axis perpendicular to its sides is the same as the moment of inertia about a diagonal. The distance c from the neutral axis to the extreme fiber in compression for a beam with the diagonal vertical, however, is $\sqrt{2}$ times as great as that for the beam with the sides vertical. If it is assumed that the fibers fail at the same stress S in the two beams of the same material, the maximum load for the beam with sides vertical would be expected to be 1.414 times that of a beam with the diagonal vertical. Tests have shown, however, that the sustained loads are practically equal. A factor of 1.414 then must be applied to the usual formula in calculating the strength of a beam with the diagonal vertical when using stress values S determined by standard tests of square specimens. Hence, such a beam of square section with the diagonal vertical has a form factor, F, equal to 1.414, and the formula becomes

$$M = 1.414 \frac{SI}{c} \quad \ldots\ldots\ldots (3)$$

For a circular section the form factor, F, has been found by a series of tests to be 1.18. In comparing a beam of circular section with one of square section, it is found that for the same area, the section modulus, I/c, of the square is approximately 118 per cent of that for the circle. The modulus of rupture of the beams of circular section, as calculated by the usual formula, was found to be about 115 per cent of that of the matched beams of 2-in. square section. This shows that a beam of circular section and one with a square section of equal area will sustain practically equal loads.

It is thus seen that form factors may be greater or less than unity, depending on the type of section. Ignoring the form factor may result in errors of calculation of as much as 50 per cent. In some cases the calculations may be over-conservative, in others, over optimistic. It is particularly important to apply form factors when calculating the strength of beams of I and box section.

In analyzing the form factor effect, it is necessary to consider the structure and characteristics of timber. The strength of wood in tension and that in compression parallel to the grain are very different, the tensile strength ranging from two to four times the compressive strength. When a beam of normal wood fails, it gives way first at the surface on the compression side, and these fibers lose some of their ability to sustain load. The adjacent fibers receive a greater stress and, with this redistribution of stress, the neutral axis moves toward the tension side and shortens the arm of the internal resisting couple, giving an increasing stress in tension. This process continues until tension failure occurs.

Whatever the exact nature of the supporting action, it appears reasonable that the least stressed fibers are free to lend the greatest supporting action and that, from the standpoint of position, the ability of fibers to lend supporting action decreases as their distance from the most highly stressed fibers is increased.

HEIGHT FACTOR OF RECTANGULAR BEAM

When the height of a beam of rectangular cross-section is increased, the modulus of rupture is decreased. Such a beam has a height factor, H, less than unity. For rectangular beams, the height factor may be expressed by the following formula:

$$H = 1 - 0.07\left(\sqrt{\frac{d}{2}} - 1\right) \quad \ldots (4)$$

where d is the depth of the beam. For specimens 2 in. deep, as used in standard tests, it may be noted that this formula gives a height factor of unity. For a beam 8 in. deep, the height factor, H, becomes 0.93; and for a beam 1 in. deep, the height factor is 1.02.

This variation of unit strength with depth of beam is in harmony with the supporting-action theory, since the deeper the beam, the less rapidly does the stress decrease from the outer side toward the neutral surface and, consequently, the less able is the material adjacent to the outer surface to render support to the most heavily stressed fibers.

HORIZONTAL SHEAR IN BEAMS

The calculation of horizontal shear in beams is another important problem where the use of the usual formula, without correction, may give misleading results with timber. While the error is on the side of safety, the result is often such as to make economical design difficult, or to provide larger sizes than necessary.

Because of the shear distortion caused in the vicinity of the bases of checks or fissures that are often present in large solid beams, the upper and lower portions of a beam act partly as two independent beams. The result is that part of the end reaction or vertical shear is resisted internally by each half of the beam independently, and only part of the end reaction is taken by the beam as a whole in accordance with the usual assumption.

An extensive series of tests, supplemented by theoretical consideration and analysis, was made to develop methods of evaluating the extent of the two-beam action, and to work out design methods. This study led to the following recommendations for design:

1. Use the ordinary shear formula and the working stresses recommended for timber.
2. In calculating the reactions for use in the ordinary shear formula, (*a*) neglect all loads within a distance equal to the height of the beam from both supports; (*b*) place the heavy concentrated moving load at a distance equal to three times the height of the beam from the support; and (*c*) then treat all loads in the usual manner.
3. If a timber does not qualify under the foregoing recommendations, which under certain conditions may be overconservative, the allowable reactions for the concentrated loads may be calculated from a more precise formula.[8]

As an illustration of the significance of modifying the method of calculating shear, the following example will be of interest. Consider a 5 by 16-in. timber with a 16-ft. span, and of a grade having a unit shear stress of 100 psi. Let us investigate the maximum single moving load this timber would accommodate by means of the various formulas.

1. By the regular shear formula, ignoring double-beam action, and placing the load adjacent to the support for maximum shear, the maximum load P is found to be 5330 lb.
2. By the recommended procedure of locating the concentrated load three times the depth of the beam from the support, the maximum load P_1 is found to be 7110 lb.
3. By using the more exact method the maximum load P_2 is found to be 7820 lb.

It may thus be seen that the newer methods available for calculating strength are of great importance in the proper design and use of timber.

THE TORSION PROBLEM

Structural members designed for pure torsion are usually made with circular, elliptical, rectangular, or other regular cross-sections that have already yielded to direct mathematical treatment as regards torsion. Members designed primarily for thrust or bending and consequently having, as a rule, an irregular section are, however, subjected to torsion also, and under certain conditions they may fail by buckling and twisting through lack of sufficient torsional rigidity. In order to design against the possibility of such failure in thin deep beams or in compression members with thin outstanding parts, it is necessary to calculate their torsional rigidity, and a method of mathematical analysis has been needed for such sections as I, T, L, and U.

From studies employing the now familiar soap-film technique, and actual torsion tests, it has been possible to conclude that the torsional rigidity of such prisms, having sections composed of rectangles, is equal to the sum of the torsional rigidities of prisms whose sections are the component rectangles, corrected by a simple additive term to take account of the increased stiffness resulting from the junctions of the rectangles.

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[8] "Form Factors and Methods of Calculating the Strength of Wooden Beams," *Engineering News-Record*, Vol. 121, Part 4, July 28, 1938, p. 115.

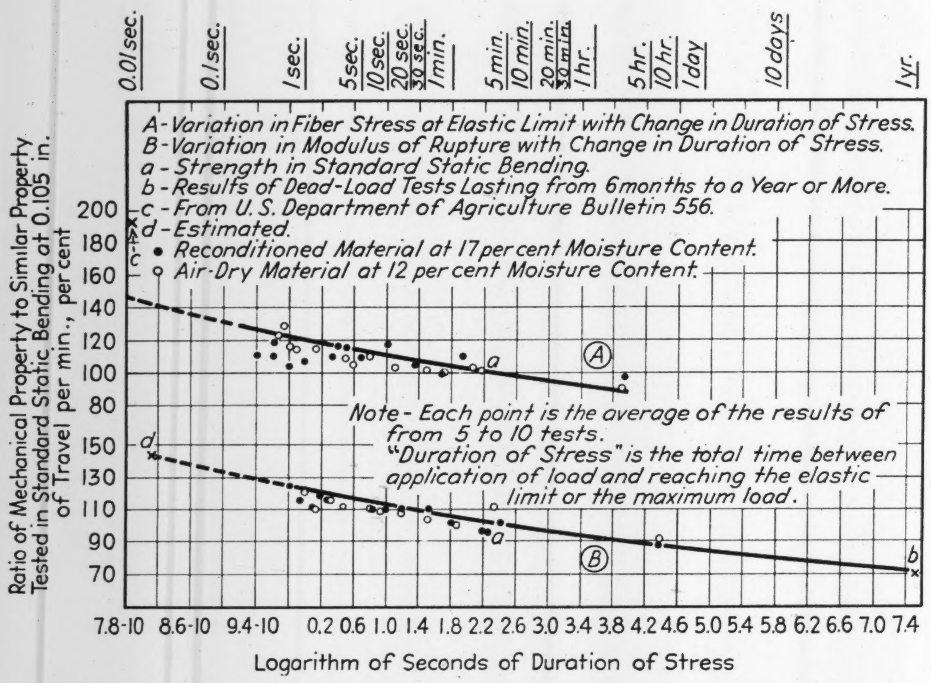

FIG. 8.—Relation Between Mechanical Property of Sitka Spruce and Duration of Stress.

These tests also show that the torsional stiffness of a beam may be materially increased by the way in which it is fastened at the ends. The difference between the moduli of rigidity of wood referred to planes radial and tangential to the annual rings may, in general, be neglected in design and a mean modulus used. For Sitka spruce this mean modulus is between one fifteenth and one sixteenth of Young's modulus parallel to the grain.

DURATION OF STRESS

The duration of stress, or the time during which a load or force acts on a beam or other wood member, has an important bearing on the use of the timber and on the adaptation of results of tests to the design of different kinds of structures or members. For instance, when an airplane traveling at high speed suddenly changes its course, as in flattening out following a dive, wood members may, without damage, be subjected for a few seconds to forces which would cause failure if applied for a longer time. In impact bending tests, where the load is suddenly applied and is maintained for but a fraction of a second, a stick will resist a force more than double that required to produce failure in a standard static bending test. On the other hand, beams under continuous loading for years, as in warehouse floors, will fail at loads one half to three fourths as great as would be required to produce failure in the standard static bending tests, where the maximum load is reached in a few minutes.

From the foregoing, it is clear that tests made under widely different conditions of loading are not comparable, and that the allowable stress in a wood beam must be determined in accordance with the loading to which it will be subjected in service. The rapidity with which the load is applied and the duration of the stress are material factors in the result.

Figure 8 presents an interpretation of some data on the influence of rate of loading from tests of small, clear specimens. A tenfold increase or decrease in the rate of loading produces approximately a 10 per cent increase or decrease, respectively, in bending strength.

In timber testing, the allowable tolerance in rate of loading is limited to ± 25 per cent of the required rate in order to keep the variation in test results from this cause within about 1 per cent.

FATIGUE

Some tests have been carried on both in the United States and in Europe to determine the effect of repeated stress and vibration, but no extended and thorough investigation has been made.

The estimated endurance limit, based on 300,000 stress cycles, using beams of circular cross-section which were rotated so that the outer fibers were stressed in tension and compression alternately at each revolution, was found to be about 29 per cent of the modulus of rupture as determined from static tests of similar specimens.

Studies made on end-loaded, rotating cantilever beams having an enlarged cross-section at the point of support demonstrated that the endurance limit varied greatly, depending on whether the change of cross-section was abrupt or gradual.

With even what is normally considered a generous fillet, the endurance limit is lowered markedly. Tests made at the Forest Products Laboratory on tapered cantilever specimens of a form to obviate changes in cross-section that would influence failure show that the endurance limit in reversed bending is approximately 25 per cent of the modulus of rupture, and that, for a stress 20 per cent greater than the endurance limit, failure occurs at not more than 2,000,000 load reversals, and in some species at less than 1,000,000 reversals.

Other tests on Sitka spruce, in which specimens of rectangular cross-section were vibrated through approximately 5,000,000 cycles, with amplitudes of vibration about one half the calculated deflection at proportional limit, indicate that the modulus of elasticity is not greatly affected by such vibration. No change in fiber stress at proportional limit and modulus of rupture could be detected from these tests, the values being about the same for the vibrated and the control specimens. Tests also indicate that the same endurance limit is obtained with vibrated specimens of rectangular cross-section as with rotated specimens of circular cross-section.

Further studies are needed to obtain more specific information on the effects of vibration and fatigue, particularly when subjected to a large number of stress cycles, and to determine the variation of these properties with different species.

STRUCTURAL USES

"Build me straight, O worthy Master!
Staunch and strong, a goodly vessel,
That shall laugh at all disaster,
And with wave and whirlwind wrestle."
—*Longfellow.*

STRUCTURAL GRADING OF TIMBER

Individual pieces of lumber, as they come from the saw, represent a wide range in quality and appearance with respect to freedom from knots, blemishes, defects, and other characteristics. Such random pieces naturally represent likewise a wide range in strength, utility, serviceability, and value. One of the obvious requirements for the orderly marketing of lumber is the establishment of grades, the function of which is to establish utility classes, so that it is possible to obtain any desired number of individual pieces generally similar in desired characteristics. Many of the grades for lumber are established on the basis of the appearance and physical characteristics of the piece, with certain uses in mind, but without particular regard for strength. Other grades are called structural, and concern themselves with classifications of features that relate to strength and strength uses. Marked improvement and unification in lumber grading resulted from the establishment of American Lumber Standards, and from the adoption by manufacturers of structural grades that permit assignment of definite working stresses.

Economy of material is obtained when all structural units are stressed to a figure approaching the allowable maximum, and this can be effected only when the material is of uniform quality. It is the function of an efficient structural timber grading specification to classify timber of any species into quality, classes, or grades by properly and adequately limiting the permissible defects and strength factors to the end that definite minimum strength is assured and that maximum efficiency in utilization is obtained.

It is not practical, and perhaps it would be too ambitious to expect, to meet this desideratum in its entirety, but the present A.S.T.M. specifications are far along in approaching this ideal.

Back of the preparation of grading rules and the assignment of working stresses must be adequate data and knowledge of the strength properties and variability of clear wood, of the strength of structural timbers containing defects, of the influence of seasoning and of moisture changes in service, of manufacturing practice, of growth conditions, and of the mechanics of tree growth. Most of these data are now available and already published.

Structural grading is accomplished from a visual examination of the timber, in which the location as well as the size of knots and other features appearing on the surfaces are evaluated. A timber free of knots on the faces would be designated as clear, and other things being equal, would carry more load than one of lower grade containing defects. Recalling how a tree grows, however, it must be kept in mind that boxed-heart timber with clear faces may and likely does contain portions of former tree branches embedded in the interior. Hence, should such a timber be resawn to smaller units, such as dimension, a lower strength grade will result. The grade of a structural timber applies to the timber of the full cross-section as originally graded. If the timber is resawn, the smaller units must be graded individually to establish their quality. (See Figs. 9 and 10.)

One of the first structural grading rules based on actual tests and incorporating all essential principles of a structural grade was that presented in *Forest Service Bulletin No. 108*, published in 1912. In these rules the beam was divided into three volumes or zones. Volume 1 comprised the lower one fourth of the height of the beam at the center half of the length, where tensile stress is most severe; volume 2, the upper one fourth of the height of the beam at the center half of the length, where compressive stress is the most severe; and volume 3, the remainder of the timber. Larger knots were allowed on the compression side of the beam, where they are less injurious than on the tension side, with the assumption that the beam would be used with this side up.

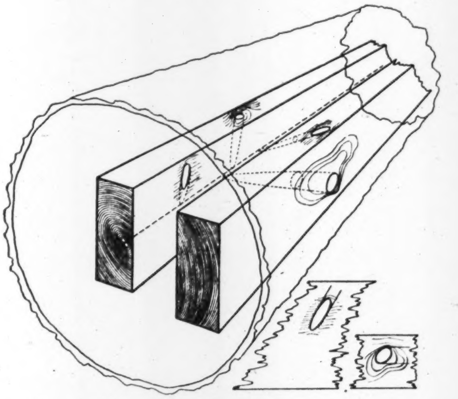

FIG. 9.—Phantom Drawing of Log Showing the Form and Shape Knots May Have When Appearing on the Different Faces of Boxed-Heart and Side-Cut Structural Timbers.

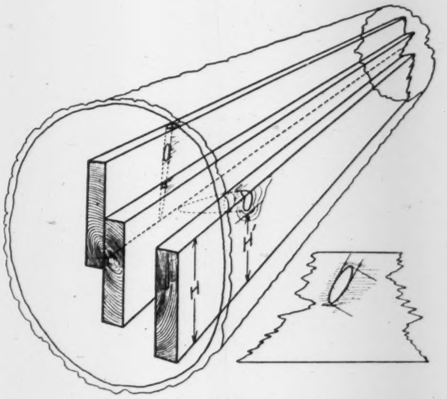

FIG. 10.—Phantom Drawing of Log Showing the Form and Shape Knots May Have When Appearing on the Different Faces of Boxed-Heart and Side-Cut Joist and Plank.

The next significant advance in structural grading was the establishment of the principles set forth in *U. S. Department of Agriculture Circular No. 295*, published in 1923. These principles were incorporated in the A.S.T.M. structural grades, sponsored by the Society's Committee D-7 on Timber, and in the structural grade examples adopted by the industry in connection with American lumber standardization, and published in Simplified Practice Recommendation R 16 – 29 of the U. S. Department of Commerce, 1929.

In 1934 structural grading received another contribution with the publication of "The Guide to the Grading of Structural Timbers and the Determination of Working Stresses," by T. R. C. Wilson. This guide is not a standard specification. It rather serves a threefold purpose of providing a technical basis for the establishment of specifications such as the present American standard, permits the industry to establish grades of any desired strength ratio, and gives the timber user a means of evaluating any material to establish the design stresses it is capable of taking.

Practical considerations that limit the ultimate theoretical efficiency of a structural grade frequently must be taken into account. For example, the compressive strength of wood is affected less by a knot of a given size than is the tensile strength. Hence larger knots may be used on the upper or compressive side of a beam with increased efficiency in utilization, but practical considerations suggest grading so that the beam can be used either side up, rather than in one definite position, the reversal of which might result in failure. Again, greater efficiency might be obtained with a more elaborate method of evaluating the size of knots, but such evaluation would be more complicated than the present simplified system, and hence less practical. A discussion of these factors is presented in a paper entitled, "Structural Timbers: Defects and Their Effect on Strength."[9]

Working Stresses

Safe working stresses are an essential corollary of structural grading. Tables of working stresses abound in engineering literature and while marked improvement in them has taken place, there is still much room for modernization and for correlating working stresses with structural grading.

Safe working stresses for timber are based both on tests of small clear specimens which afford both average values of the inherent strength of the different species and a measure of variability and on tests of large timbers which establish the influence of defects. The determination of safe working stresses involves further the adjustment of test values to meet the conditions of service. This adjustment requires consideration of (*a*) the loss in strength from defects, (*b*) the effect of long-continued loads, (*c*) the variability of individual pieces from the average, (*d*) the possibility of slight accidental overloading, and (*e*) species characteristics.

The effect of these factors is to necessitate a lower strength value for practical use conditions than the average value from tests of small clear specimens. Their combined effect may be embodied in a single factor, sometimes erroneously called a "factor of safety," which can be applied to averages from tests on small clear specimens to obtain safe working stresses. It is evident that the larger part of this factor is required to correlate laboratory test results with actual conditions of use, and only a small part may be considered a true factor of safety.

The belief that a timber with a so-called "factor of safety" of 3 or 4 will carry three or four times the load for which it is designed is a misconception. As a specific example, the average modulus of rupture of green Sitka spruce, based on tests of small clear specimens free of defects, is 5760 psi. and the basic working stress for the extreme fiber in bending for a grade of material that permits defects that reduce the strength by one fourth is 1100 psi., which shows a factor of $5\frac{1}{4}$. This factor of $5\frac{1}{4}$ is not a "factor of safety," but consists of successive reductions of one fourth for defects, one fourth for variability, seven sixteenths for long-time loading, and two fifths to provide an actual factor of safety of $1\frac{2}{3}$.

The structural grades are sometimes designated by the working stresses they are accorded, by the kind of timber according to use (joist, plank, etc.), and by the species name. Within limits, it is possible to set up grades for any species to take any desired stress by appropriately varying the defects permitted to meet any desired grade-strength ratio. While the standard establishes a variety of stress grades for the more important species, it could be extended at will to cover other species and other grade-strength ratios.

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[9] J. A. Newlin and R. P. A. Johnson, "Structural Timbers: Defects and Their Influence on Strength," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 975 (1924).

It should be noted that the stresses for the various A.S.T.M. structural grades apply to timbers used in continually dry locations or under other conditions where deterioration is not expected. Higher initial strength than would otherwise be required is desirable in timbers exposed to decay or other deterioration and to delay entailed by replacement. It may be provided by arbitrarily increasing the size of timbers or by using lower design stresses.

MODERN CONNECTORS

When timber connectors were introduced into the United States in 1930 through the National Committee on Wood Utilization, their possibilities were quickly recognized. Strength tests undertaken at the Forest Products Laboratory established the most promising types and furnished basic design data for use under optimum conditions. Simultaneously, commercial development by the National Lumber Manufacturers Association occurred.

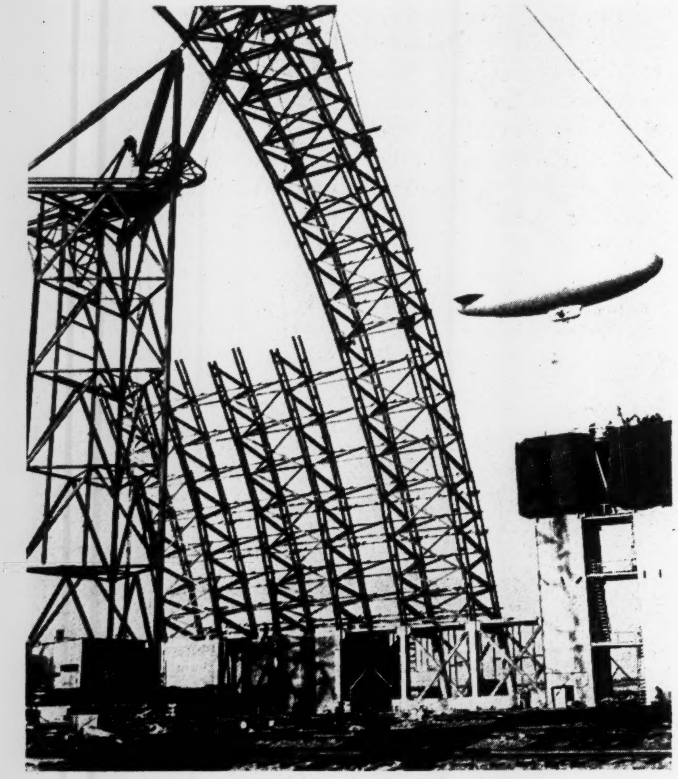
The use of timber connectors by the construction industry has been effective in introducing substantial savings. In the short period between 1933 and 1942 there were built over 100,000 structures in which modern connectors were used, employing about 5 billion board feet of lumber valued at $165,000,000. The amount of lumber in these structures used directly with the connectors approximated 925 million feet, valued at over $30,000,000.

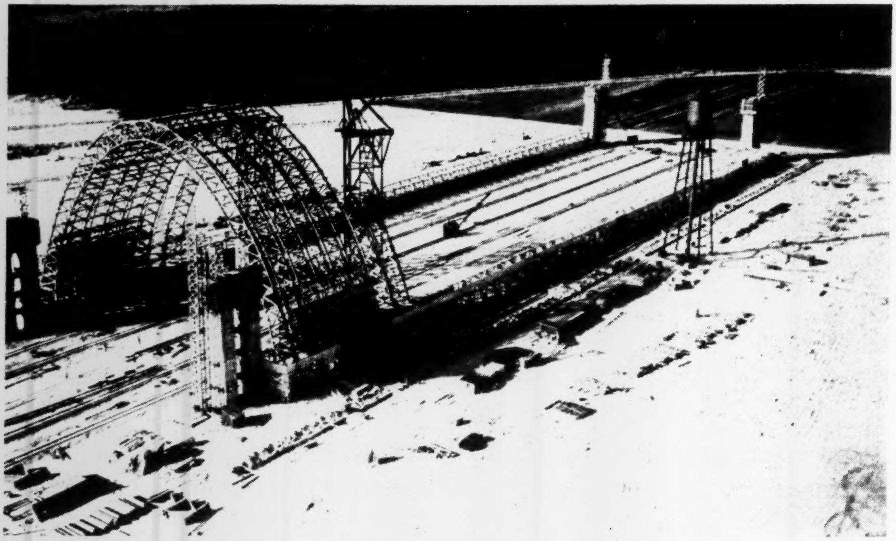
Connectors are devices used in conjunction with bolts to increase the efficiency of joints in wood members. The split-ring type fits into precut grooves; the alligator type is pressed into the wood between the members to be joined; other types fit into bored recesses. Whatever the type, they all serve to reinforce the joint by providing effective shear resistance between the members.

More recent tests have been made at the Forest Products Laboratory to determine the strength of several of the connector types in a range of sizes, applied in various ways, when used with different species of wood. A number of other design details were also investi-

Three-Hinged Arch Highway Bridge, Umpqua National Forest, Oregon.

Illustrative of the many outside structures using timber connectors in conjunction with creosote-treated timbers to insure long life. The span is 135 ft.





The Timber Trusses Used By the Navy for Blimp Hangars Are a Striking Example of Connector Construction.

The clear roof span is 237 ft.; the height 153 ft.; and the length of the structure 1000 ft.

gated. The results are now being prepared for publication.

Modern metal connectors have been used in a great variety of structures and structural members. These include timber trusses of 200-ft. span, such as were used in buildings of the Golden Gate International Exposition; timber bridges of various kinds; and radio and forest lookout towers.

When durability or decay resistance is needed in structures using connectors, it may readily be obtained by selecting species of high natural decay resistance or by preservative treatment. Lookout towers are commonly made of Douglas fir or southern yellow pine. The timbers are first completely shop-fabricated for assembly on the site, including the boring of the necessary bolt holes and the making of the grooves for the ring connectors. After fabrication, the timbers are treated with a wood preservative. This treatment insures effective penetration of the preservative in the bolt holes and ring grooves. Long service is assured in such treated structures for exterior use.

GLUED LAMINATED CONSTRUCTION

Laminated construction refers to two or more layers of wood glued together with the grain of all layers approximately parallel. The laminations may vary as to species, number, size, shape, and thickness. The properties of laminated members are essentially the same as those of solid wood, but laminated members, if well constructed, are usually more uniform in strength properties and less apt to change shape with variations in moisture content.

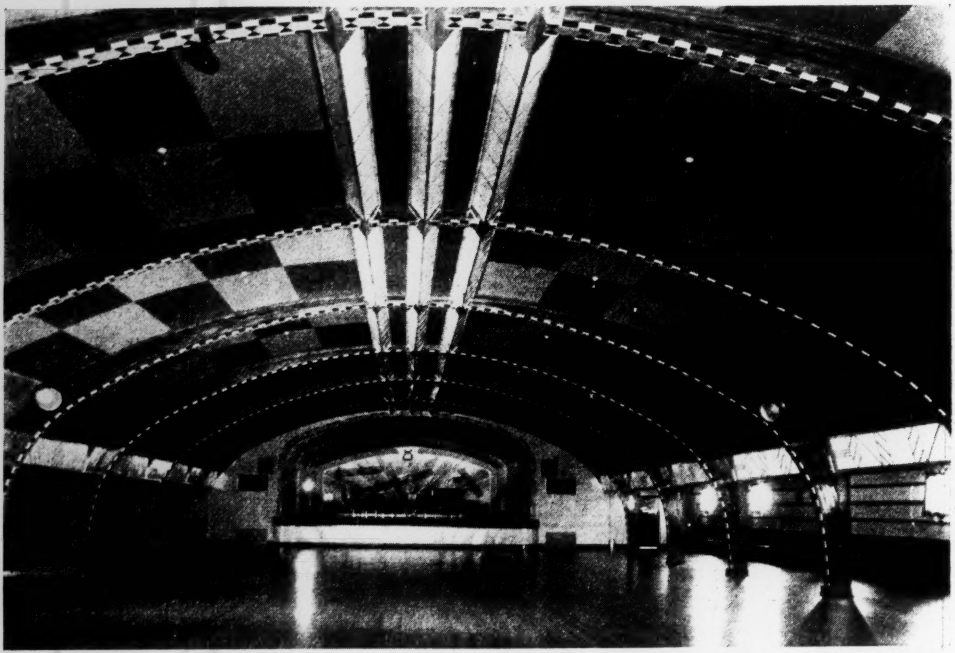
Laminated Arches:

A recent development in timber structures is the use of laminated members made in straight or curved form by bonding seasoned and properly surfaced boards with a water-resistant glue. A principal application is the construction of wood roof arches which have greater strength and efficiency and better appearance than were previously possible. The first building employing this type of construction in the United States was erected in 1935 and is 45 ft. wide with bent and tapered members that follow side wall and roof slope from foundation to ridge. More notable is the recent use of three-hinged wood arches formed by this method to span 120 ft. between buttressed side walls of a college field house. Glued, laminated wood members have been used in hundreds of structures, including gymnasiums, community buildings, churches, theaters, and clubhouses, and their use is expected to expand as the possibilities of this form of construction become more widely known.

Back of this development have been several years of research at the Forest Products Laboratory. Technical problems investigated included the effect of initial stress resulting from bending the laminae to curved form and the allowance therefor which must be made in design; the practical limitations of severity of curvature; the possibility of using up to 60 per cent of low-grade material in interior laminations; the type of scarf joint necessary for the outer laminations; and similar design and construction problems. The results of this extensive study, as well as recommendations for design stresses and fabrication practice, are presented in a recently issued publication, U. S. Department of Agriculture Technical Bulletin No. 591, entitled "The Glued Laminated Wooden Arch."

COMPOSITE CONSTRUCTION

A composite construction, in which a creosoted timber base is overlaid with a lightly reinforced concrete mat, has been



Laminated Wood Arch Construction Provides Attractive Buildings with Large Unobstructed Floor Areas.

This span is 53 ft., but spans of over 300 ft. have been employed.

Footbridge Over the Yahara River, Madison, Wis. Employing Laminated Wood Arch Construction.

As one of the first outside installations, a number of special features are incorporated to provide increased serviceability. The span is 73 ft.

successfully employed in bridge decks and for other heavy duty floors for some years. Special triangular shear keys are employed to secure adequate bond of the two materials. Advantages are that joist and stringer systems are not required, and that the timber base, which can be simply laid, provides the required tensile strength, and during construction serves effectively in facilitating the remainder of the work.

HOUSING

Considerable research has already been conducted on various problems relating to house construction with the object of reducing initial costs and maintenance and improving serviceability. Included have been an analysis of the strength and rigidity of frame walls, showing the importance of diagonal bracing; studies of the condensation of moisture in walls, and the effectiveness of vapor barriers; tests of fire-resistance of structural units; and research on painting problems.

Some experimental work also has been done in the development of unit construction or so-called prefabricated construction. One such system employs the use of plywood as a skin covering, glued to stringers to form lightweight structural units. In this construction, the plywood acts as a stressed covering and functions to provide an effective structural distribution of the material. This system has already been used to a considerable extent commercially.

Another development in unit construction employs small panels that can be fabricated with ease from short length lumber in small woodworking plants, and lends itself to quick erection.

RIGID FRAMES

A form of structural unit gaining favor is the so-called "rigid frame." This consists essentially of a wood frame covered with a sheet material, usually plywood. The sheet material may be nailed to the frame or may be bonded with casein water-resistant glue. Casein glue has the advantage that it can be applied cold and that it does not need to be applied immediately after mixing. After the application of the glue, the cover is nailed to the frame. The principal function of the nails in this case is to apply suitable pressure while the glue sets. Nailed rigid frames were used successfully at the Great Lakes Exposition buildings in Cleveland a few years ago.

Obviously, the stress analysis of rigid frames is complicated. So far, but little research has been done to demonstrate their strength and rigidity and to develop design methods.

BARREL ROOFS

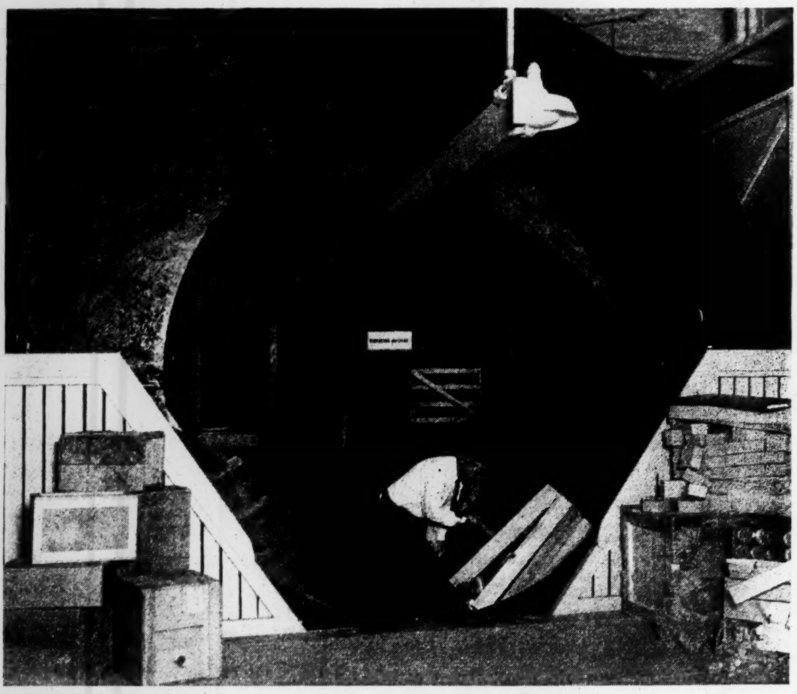
A construction with interesting possibilities is the barrel roof. A relatively thin membrane used in a sharply curved form gives an arch effect which is economical of material. Barrel roofs formed from concrete have already been used in the United States, and there is some possibility of using wood for this kind of construction, but little research work has so far been done in working out design details.

SHIPPING CONTAINERS

Amid the glamour of production lines and the impressive parade of war machines and other products, the all-important function of the shipping container is apt to be overlooked. Yet to wood and fiberboard has fallen the important task of protecting most of the nation's production while in transit. As was rediscovered during the early days of the war, when many products were damaged in shipment, production in itself is of no avail if the things produced do not safely reach their destination. An efficient shipping container is one that delivers its contents to its destination in a satisfactory condition and at a minimum cost.

A box or crate constitutes a complicated structure that does not lend itself satisfactorily to stress analysis, so that developmental work must be carried on by laboratory testing methods. Active research on shipping containers began many years ago with the invention of the hexagonal testing drum equipped with interior hazards, by means of which the container is continually shifted in accordance with a fairly definite cycle of falls. Several decades of study have established basic principles of construction for various forms of wood boxes and crates and fiber containers, the commercial application of which has resulted in reduced loss and damage claims and great savings in materials.

Present war and domestic shipments require approximately 12 billion board feet of lumber annually, in addition to large quantities of fiberboard and the new water-resistant fiberboard or "V" board. Aside from the direct function of improving the serviceability of containers, current container research on war products has effected, as a byproduct, a saving in shipping space of approximately 20 per cent, which is important enough as an objective in itself. This means that, on the average, four ships can now carry the material which formerly required five. Estimated savings in shipping costs of $50,000,000, not including railroad and truck savings, have been reported.



About One Third of the Annual Lumber Cut in 1942 Was Used for Boxes and Crates.

The hexagonal-drum box testing machine, or mechanical baggageman, was designed at the Forest Products Laboratory to study the weaknesses and improve the design of shipping containers.

This war calls for particular care in the shipment of weapons. In the first World War, war machines were shipped to French docks; today, heavy but delicate equipment of all types may be landed on rocky beaches or towed hrough rough surf. Warehouses or even adequate cover are frequently lacking and thus the container serves as warehouse, also, often for long periods of time.

Application of the principles of container design that are now being employed so effectively should be of great assistance in connection with postwar world trade.

MODIFICATION OF PROPERTIES

> "The witches assembled and started anew,
> To mix a potion, a better brew;
> Loosed was lightening, and a thunder blast;
> Strange the product—their dreams surpassed."
> —*Anon.*

GLUES

Perhaps no single factor has been more responsible for the significant advance in the field of modern wood construction than the development in glues and gluing technique. The past decade has brought synthetic resins that have greatly widened the horizon for fabricated products that must resist continued exposure to moisture changes. Advance with other glues has also been important.

During the first World War there were available starch glues, hide glue, fish glue, casein glue, and blood glue, the latter two being somewhat water-resistant. Research has been concerned with the determination and comparison of the properties of glues, the relation between chemical and physical properties, performance under various service conditions, establishment of conditions essential to securing good glued joints, and development of specification tests and requirements to establish maintenance of quality of acceptable glues. This has led to the evaluation of the serviceableness or durability of glued joints by various means, taking into account different exposures, and exposure cycles, and temperatures and moisture variations.

Casein Glues:

The forms, characteristics, and properties of water-resistant casein glues have remained substantially the same for many years. The dried casein of milk is the basic constituent of this class of glues. It is combined with alkalies or alkali-producing chemicals which, when mixed with water, dissolve the casein. The addition of lime or other materials causes the glue to set and later retain a part of its strength, even when saturated with water. Toxic materials are frequently added to make the set glue more resistant to molds and other deteriorating organisms.

Among numerous other applications, casein glues are still used to a considerable extent in aircraft construction although they are gradually being replaced by resin glues in this field. They have been in use for many years and the details of their preparation and application are generally known.

Blood-Albumin Glues:

Previous to the development of the synthetic-resin glues, the blood albumins were the most water-resistant and durable of the woodworking glues. Blood-albumin glues are formulated by the user, usually from dried animal blood. The most water-resistant glues of this type are a combination of blood and formaldehyde. Hot pressing is necessary, however, to develop their full strength. The use of blood albumin at present in woodworking glues is confined chiefly to combinations with resins to produce glues of special operating characteristics, to augment existing supplies of resins, or to cheapen the resin glues.

Synthetic-Resin Glues:

The best-known and most commonly used synthetic-resin glues are the phenol-formaldehyde and urea-formaldehyde types. There are also some synthetic-resin adhesives of the melamine, resorcinol, and polyvinyl-ester types, but they are as yet not widely used. The phenol and urea resins may also be combined with each other, with other resins, or with other chemicals to form glues of somewhat different basic characteristics than glues made from either resin alone. The phenols are also mixed with blood albumin and walnut-shell flour; or other materials may be added to any of the resin glues. As a result of this practice, there are resin glues with varying amounts of other materials, some of which are variously referred to as "fortified," "modified," and "extended."

Synthetic-resin glues may be classified as thermosetting or thermoplastic. The phenol, urea, melamine, and similar resins belong to the thermosetting class and, once the condensation reaction, which is hastened by heating, is complete, no subsequent softening occurs even though the temperature is increased beyond the original setting temperature. Synthetic resins of the thermoplastic class, such as vinyl acetate or butyrate, soften whenever the temperature is raised above the softening range that is characteristic of each particular type of resin. A thermoplastic resin must be first heated and then cooled under pressure in using it as an adhesive.

TABLE X.—CURING TIME, TEMPERATURE, AND PRESSURE[a] REQUIREMENTS FOR VARIOUS TYPES OF GLUES FOR BONDING WOOD.

Type of glue	Classification	Approximate Time[b]	Approximate Temperature, deg. Fahr.[b]
Phenolic resin	Hot press	3 to 6 min.	250 to 300
	Low temperature	[c]	[c]
Urea resin	Hot press	3 to 6 min.	220 to 260
	Cold press	6 to 16 hr.	Not less than 70
Casein	Cold press	4 to 12 hr.	Room temperature
Blood	Hot press	2 to 5 min.	180 to 300
Vegetable protein (soybean)	Cold press	4 to 12 hr.	Room temperature

[a] Recommended approximate pressures range from 100 to 200 psi. for flat panels, but some of these glues may be used also for bag molding and in nailed glue assemblies with pressure as low as 50 psi. or less.

[b] Curing time and temperature figures are at the glue line. Allowance must be made for the time it takes to bring a glue line up to the specified temperature.

[c] Time and temperature requirements for low temperature phenolics are not yet completely defined.

Synthetic-resin glues may be marketed in the form of dry film, dry powder, suspension or solution in water, or non-aqueous solution. A number of them are formulated for hot pressing, during which the high temperatures soften the resin to a flowing or plastic condition and complete the setting reaction. Resins now in common use which set at a lower temperature are mainly of the urea-resin type and contain catalysts which accelerate the chemical reaction. The setting temperatures of such glues are 70 F. or higher.

Table X presents briefly the requirements of the various glues as to temperature and pressure in forming a bond with the wood.

Glue Performance:

One of the accelerated tests employed to determine the durability of glued joints involves a soaking-drying cycle (2 days in water followed by 12 days in air at 30 per cent relative humidity) to simulate extreme conditions of service. Table XI presents a comparison of the various glues under this test when used in plywood.

It may be noted from Table XI that the starch and animal glues lose all their strength at the first soaking. The casein glue is much more water-resistant and is admirable for many use conditions involving mild exposure, but at the end of 2 yr. of this severe test it had lost its strength. The blood glue is losing its strength toward the end of the 4 yr. or 100 complete cycles of accelerated testing, the failure of the samples being in the glue. The hot-pressed, phenolic-resin glue is still maintaining a high standard of strength and a high percentage of wood failure at the end of 6 yr., which encourages great confidence in its possibilities for long service when used with plywood under severe exposure conditions. The tests on the urea resins have not been under way long enough to give the 6-yr. comparison. It has been observed, however, that while joints made with the hot-pressed urea glues are very water-resistant at ordinary temperatures, they do not appear to withstand exposure to hot water and they weaken slowly at dry high temperatures.

Considering the "cold press" resins, which set at lower temperatures, the phenolic-resin type, after being heated to about 150 to 200 F. for some 4 hr. subsequent to pressing to develop the full strength and durability of the joint, appears to give results approximately equal to those produced by the hot-press method. In the tests the cold-press urea glues were definitely more resistant than the casein glues, but, as far as present data go, they lacked the durability under all conditions of exposure that has characterized the well-made phenolic glue joints.

TABLE XI.—COMPARISON OF GLUES UNDER ACCELERATED TESTS.
3/16 in., three-ply yellow birch plywood: cycle: 2 days soaking. 12 days drying.

Kind of Glue	Loss in Strength of Glued Joints			
	Soaked and Tested Soon After Gluing	Tested After 2 yr.	Tested After 4 yr.	Tested After 6 yr.
Animal	Complete	....	....	....
Vegetable (starch)	Complete	....	....	....
Vegetable protein	Slight to complete, depending on species	....	....	....
Casein	Slight	Complete	....	....
Blood	None	Moderate	Marked	Marked
Urea resin (hot pressed)	None	Slight[a]	Moderate	....
Phenolic resin (hot pressed)	None	Slight[a]	Slight[a]	Moderate

[a] Slight decrease in test values may be due to deterioration of wood.

Electrostatic Heating:

The application of high frequency to the heating of wood is receiving much attention, and in some applications is past the laboratory stage. Electrostatic heating provides a ready means of quickly heating wood even in large sizes, and has the significant advantage of heating the interior without relying on conduction from the surface.

It is applicable, also, to the seasoning of wood, but at present does not appear to be so practical for this purpose. Commercial applications in connection with the gluing of plywood have already been in practice where a hot-press operation is required.

The method holds great promise for the use of hot-setting synthetic resins for various laminated products, such as arches, beams, and ship timbers, and particularly where large members are involved. However, extensive research is still required to determine its advantages and limitations in various applications.

PLYWOOD

In plywood, the orthotropic structure of wood is further diversified by a reorientation of the material. Plywood consists of a combination of three or more sheets of veneer, with the grain of alternate plies usually at right angles. The outside plies are called faces and the center ply the core. In plywood of five or more plies, alternate intervening plies between the faces and core, whose grain direction is at right angles to that of the face plies, are called cross bands.

Because plywood can be made with any desired number of plies from veneer of any desired thickness and species or combinations of species, it presents a versatility that affords almost unlimited choice in design for special purposes. Further diversification can be obtained by orienting the grain of adjacent plies, not only at right angles but at various angles. In this way a material can be constructed to meet special design needs for aircraft and other uses.

Arrangement of Plies:

The tendency of cross-banded products to warp with moisture changes is largely eliminated by balanced construction. This consists of arranging the plies in pairs about the core so that for each ply there is an opposite, similar, and parallel ply. Matching the plies involves a consideration of (1) thickness, (2) kind of wood with particular reference to shrinkage and density, (3) moisture content at the time of gluing, and (4) angle or relative direction of the grain.

Balanced construction hence requires the use of an odd number of plies. If only two plies are glued together with the grain at right angles to each other, each ply tends to distort the other when moisture content changes occur, and cupping usually results. Similar results are likely when any even number of plies is used. The use of balanced construction is highly desirable in plywood and is especially important in thin panels that must remain flat.

Strength and Shrinkage of Plywood:

As compared with solid wood, the chief advantages of plywood are an equalization of strength properties along the length and width of the panel, greater resistance to checking and splitting, and less change in dimensions with changes in moisture content.

Itshould be noted that, in reorienting the direction of the grain of the veneer to make plywood, the strength of wood in one direction is improved at the expense of that in the other direction.

The greater the number of plies for a given thickness, the more nearly equal are the strength properties in the two directions of the panel and the greater is the resistance to splitting.

The shrinkage of plywood varies with the species, the ratios of ply thicknesses, the number of plies, and the combination of species. Plywood is relatively stable in dimension in the length and width direction of the panel under moisture changes. The average shrinkage values, from a soaked to an oven-dry condition, for three-ply panels having all plies in any one panel of the same thickness and species was about 0.45 per cent parallel to the face grain and 0.67 per cent perpendicular to the face grain, with ranges of from 0.2 to 1 per cent and 0.3 to 1.2

TABLE XII.—DESIGN METHOD AND ALLOWABLE STRESSES.[a]

Property	Direction of Stress with Respect to Direction of Face Grain	Portion of Cross-Sectional Area To Be Considered	Unit Stress To Be Used
Tension	Parallel or perpendicular	Parallel plies[b] only	Unit stress for extreme fiber in bending
	±45°	Full cross-sectional area	One fourth unit stress for extreme fiber in bending
Compression	Parallel or perpendicular	Parallel plies[b] only	Unit stress in compression parallel to grain
	±45°	Full cross-sectional area	One third unit stress in compression parallel to grain
Shear	Parallel or perpendicular	Full cross-sectional area	Double unit stress for horizontal shear
	±45°	Full cross-sectional area	Four times unit stress for horizontal shear
Shear in plane of plies	Parallel, perpendicular, or ±45°	Full shear area	1. Conditions without stress concentration. A. Joints between plies in plywood panels acting as a beam—one half unit stress for horizontal shear 2. Conditions with stress concentration. A. Symmetrical concentration. Joints in panels with stressed plywood covers, for *interior joists* with end headers, or without end headers if ratio of joist depth to joist width does not exceed 2—one-half unit stress for horizontal shear B. Unsymmetrical concentration. Joints in I and box beams with plywood webs; and joints in panels with stressed plywood covers, for *exterior* joists with end headers, or without end headers if ratio of joist depth to joist width does not exceed 2—one fourth unit stress for horizontal shear
Load in bending	Parallel or perpendicular	Bending moment $M = KSI/c$ where S = unit stress for extreme fiber in bending; I = moment of inertia computed on basis of parallel plies only; c = distance from neutral axis to outer fiber of outermost ply having its grain in the direction of the span; $K = 1.50$ for three-ply plywood having the grain of the outer plies perpendicular to the span; $K = 0.85$ for all other plywood	Unit stress for extreme fiber in bending
Deflection in bending	Parallel or perpendicular	Deflection may be calculated by the usual formulas, taking as the moment of inertia that of the parallel plies plus one twentieth that of the perpendicular plies. (When face plies are parallel, the calculation may be simplified, with but little error, by taking the moment of inertia as that of the parallel plies only.)	Unit value for modulus of elasticity
Deformation in tension or compression	Parallel or perpendicular	Parallel plies[b] only	Unit value for modulus of elasticity
Bearing at right angles to plane of plywood		Loaded area	Unit stress in compression perpendicular to grain

[a] The suggested simplified methods of calculation apply reasonably well with usual plywood types under ordinary conditions of service. It is recognized, however, that they are not entirely valid for all types of plywood and plywood constructions, or for all spans and span-depth ratios.

[b] By "parallel plies" is meant those plies whose grain direction is parallel to the direction of principal stress.

per cent, respectively. The panels tested ranged in thickness from $\frac{1}{10}$ to $\frac{1}{2}$ in. In general, the shrinkage of plywood with five or more plies is somewhat less than that of three-ply material and more nearly equal in directions parallel and perpendicular to the face grain.

Design Methods for Plywood:

Extensive tests at the Forest Products Laboratory have resulted in the evaluation of the properties of many types of plywood and the formulation of allowable stresses and design methods. These are briefly summarized in Tables XII, XIII, and XIV.

The tremendous expansion in the aircraft program has again brought wood, and particularly plywood, into extensive use for such constructions. Application of these materials to present-day designs calls for detailed information on their behavior in complicated sections and under unusual conditions of stress. Exhaustive studies have been made and are being continued to supply these design criteria. Today such data must be restricted in distribution; tomorrow they will be used to provide better, cheaper, and more pleasing homes, furniture, planes, cars, and other articles of commerce.

TABLE XIII.—BASIC STRESSES FOR DOUGLAS FIR (COAST REGION) FREE FROM DEFECTS.[a]

Extreme fiber in bending, psi	2 000
Compression perpendicular to grain, psi	325
Compression parallel to grain, psi	1 466
Maximum horizontal shear, psi	120
Modulus of elasticity, psi	1 600 000

[a] Basic stresses for some other species are given in Table VIII. "Guide to the Grading of Structural Timbers," U. S. Department of Agriculture, *Miscellaneous Publication, No. 185.*

Appropriate unit stress for Table XII may be obtained from Table XIII by multiplying the basic values by the appropriate reduction factor, as follows:

1. For Douglas fir plywood to be used in dry locations (moisture content 16 per cent or less) the basic stresses for extreme fiber in bending, compression perpendicular to grain, and compression parallel to grain may be increased by 25 per cent. (No increase for maximum horizontal shear or modulus of elasticity.)

2. The basic stresses are for clear wood without defects. An appropriate reduction factor is to be used according to the estimated grade of material with respect to defects allowed. When defects present are estimated to reduce the strength one fourth, multiply the basic stresses by three fourths, etc.

TABLE XIV.—EXAMPLE OF UNIT STRESSES FOR DOUGLAS FIR PLYWOOD OF THREE-FOURTHS GRADE TO BE USED IN DRY LOCATIONS, FOR USE WITH TABLE XII.

Property	Direction of Stress With Respect to Face Grain	Unit Stress, psi.
Tension	Parallel or perpendicular	1 875
	±45°	470
Compression	Parallel or perpendicular	1 375
	±45°	460
Shear	Parallel or perpendicular	180
	±45°	360
Shear in plane of plies	....	22[a]
Bending	Parallel or perpendicular	1 875
Modulus of elasticity	Parallel or perpendicular	1 600 000
Compression perpendicular to grain (bearing)	....	400

[a] Applies to shear stresses, such as in I and box beams, where the stress is concentrated at the inner edge of the flange. For other conditions as outlined in Table XII, different values would be found as calculated by the appropriate factor.

Example: What unit stress should be used for tension parallel to face grain for plywood of a three-fourths grade to be used in dry locations?

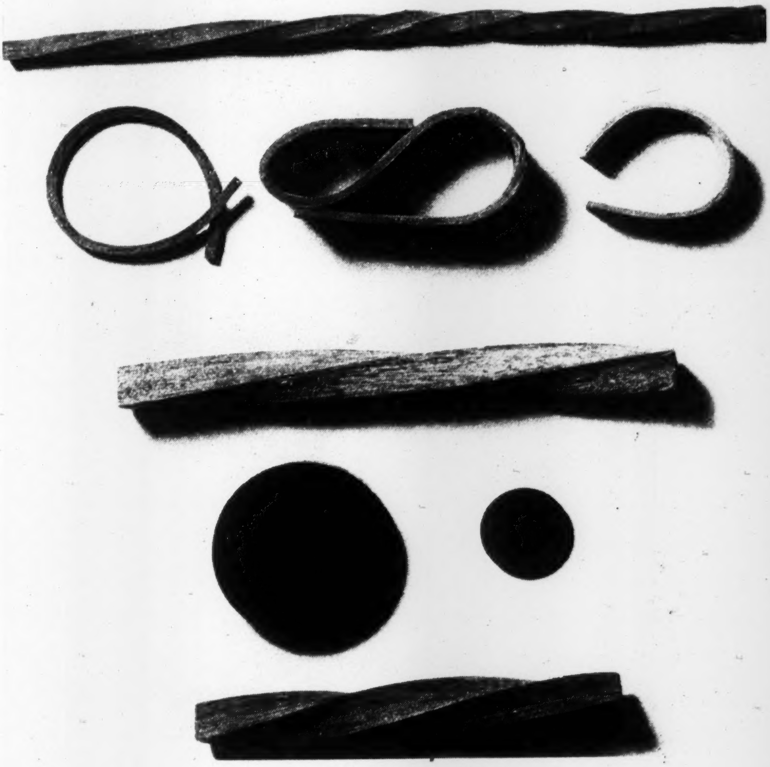
Procedure: 2000 × 1.25 × 0.75 = 1875 psi.

NEW PRODUCTS FOR SPECIAL USES

Aside from the use of wood and plywood in a more or less natural form, five other types of products should be considered as engineering materials: (1) resin-impregnated wood (compressed or uncompressed); (2) wood plastic; (3)

laminated paper plastic; (4) heat-stabilized, compressed wood; and (5) insulation and building board.

Some of these products are relatively new, and the many diverse applications already presage extended future use.



Samples of Plasticized Oak.

The figure 8-shaped sample was bent with the grain; the open loop to the right was bent across the grain; the large disk was laminated in pressing from small pieces; and the small disk was made from blackjack oak sawdust.

Resin-Impregnated Wood:

When wood is impregnated with synthetic resin and subjected to heat, new properties are imparted to the material. When unpolymerized resin-forming materials of the phenol-formaldehyde type, in contrast to preformed resins, are used for the treatment, the product is made highly resistant to swelling and shrinking. This is due to the fact that the resin-forming materials enter the cell-wall structure and become chemically combined throughout the cell wall. This treatment also gives a marked improvement in some mechanical properties, particularly hardness and compression perpendicular to the grain. Indications are that the acid and decay resistance are also improved.

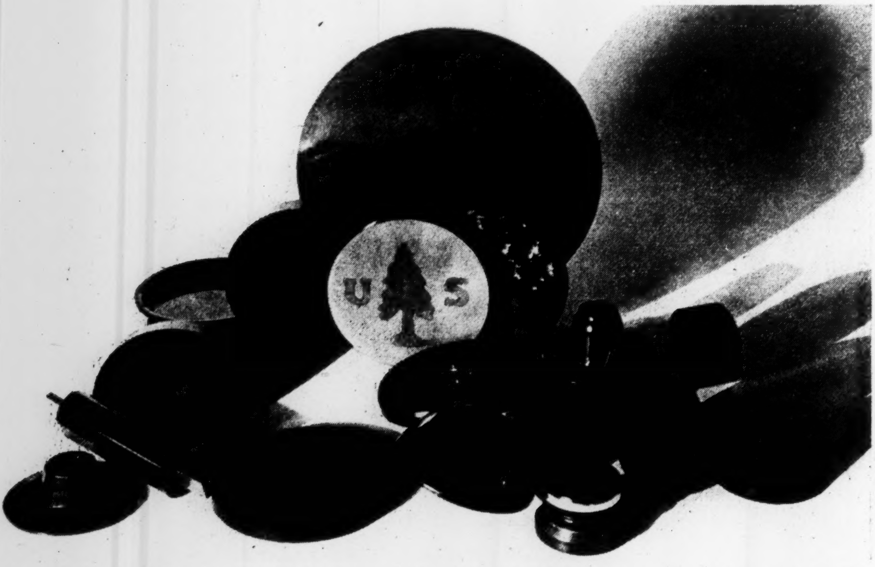
With plywood or laminated wood, either the outer ply only may be impregnated or all plies may be treated. An effective surface is provided that greatly reduces weathering and checking when exposed and may possibly serve in lieu of paint for exterior use.

When veneer is impregnated with

synthetic resin-forming materials and made up into the form of plywood or laminated constructions, the material may be compressed at the same time the resin is set. The resin-forming materials permit bonding without the addition of glue or other adhesives, and also permit the plies to be compressed at considerably lower pressures. The resulting product is a dense material having greatly improved mechanical properties. For compregnated spruce, having a specific gravity of 1.32, tests have shown a modulus of rupture of 43,400 psi., a tensile strength parallel to the grain of 42,500 psi., and a modulus of elasticity of 4,418,000 psi.

Composite material with resin-treated compressed faces on either an untreated or a resin-treated precured core can be made up in a single compressing and assembly operation. The faces are plasticized by the resin-forming chemicals to the degree that they may be considerably compressed under the low assembly pressures (200 to 250 psi.) which will cause only a slight compression of the core. A material is thus obtained with a smooth, hard, shell-like exterior surface and a normal wood interior. In this way, a greatly improved surface hardness and strength may be obtained with but little increase in weight.

Resin-impregnated, compressed, laminated wood has already been used for airplane propellers. Sometimes compressed wood is used near the hub while normal wood is spliced on near the tip, the object being to use each where it is most efficient. Such propellers may be used effectively with metal hubs of variable pitch.



A Variety of Molded Shapes Made Experimentally from the Forest Products Laboratory's Waste-Lignin Molding Powder.

Wood Plastic:

The Laboratory has developed from sawdust or other wood waste a plastic molding compound, useful in nonstressed or moderately stressed articles. In the method used, hardwood sawdust is treated by a dilute acid under steam pressure. The residue, after removal of the sugar solution formed by this process, is ground to a powder and, when mixed with a plasticizer, can be molded into various shapes under proper temperature and pressure. The result is a cheap molding compound that forms into a black lustrous plastic. It is relatively hard and strong, has high electrical resistance, and can be cut with machine tools like metal. It bonds satisfactorily to metals such as brass, bronze, and aluminum. Because of its light weight and high acid resistance, it has been used in storage battery boxes. The wood plastic requires up to 50 per cent less critical war-restricted resins than do the general-purpose phenolics using wood flour.

TABLE XV.—PHYSICAL PROPERTIES OF LAMINATED PAPER.

All tests made according to proposed Federal Specification for Testing Organic Plastics dated July 7, 1942.

Tests	Parallel Laminated	Cross Laminated
Molding conditions:		
Pressure, psi	250	250
Temperature, deg. Fahr.	325	325
Time (thin layers), min.	12	12
Specific gravity	1.35 to 1.40	1.35 to 1.40
Tensile properties, psi.:		
Tensile strength, with grain	36 000	25 000
Tensile strength, across grain	18 000	....
Modulus of elasticity, with grain	3 000 000	2 200 000
Modulus of elasticity, across grain	1 500 000	....
Compressive strength, psi.:		
Edgewise with grain	19 000	18 000
Edgewise across grain	17 000	....
Flatwise	40 000	40 000
Flexure, psi.:		
Modulus of rupture, with grain	32 000	26 000
Modulus of rupture, across grain	20 000	....
Modulus of elasticity, with grain	3 000 000	2 000 000
Modulus of elasticity, across grain	1 500 000	....
Shear, Johnson double shear, psi.:		
Edgewise, across grain	15 000	14 000
Edgewise, with grain	13 000	....
Flatwise, across grain	15 000	14 000
Flatwise, with grain	13 000	....
Izod impact. ft-lb. per inch of notch:		
Lengthwise specimen, notched flatwise	5.0	4.0
Lengthwise specimen, notched edgewise	0.8	0.7
Crosswise specimen, notched flatwise	2.0	....
Crosswise specimen, notched edgewise	0.6	....
Rockwell hardness	M 100	M 100
Water absorption, per cent:		
⅛ by 1 by 3-in. specimen, 24 hr.	2 to 4	2 to 4

In addition to the wood plastic, a resin-impregnated, hydrolized wood sheet can be laminated under heat and pressure to form, under double curvature if desired, a material that has promise for many uses. The properties vary with the processing details. Representative strength properties are approximately as follows:

Specific gravity	1.40
Modulus of rupture, psi	9 000 to 11 000
Tensile strength, psi	7 000
Modulus of elasticity, psi	900 000
Izod impact, ft-lb. per inch of notch	0.5

Laminated Paper Plastic:

An improved paper-base plastic of high tensile strength has recently been developed and is already being experimentally used in the form of aircraft parts, including wing ribs, wing tip skins, and control surfaces. Experimental data thus far indicate that the product has very high tensile strength, that it can be molded to desired shapes at temperatures and pressures and on equipment now used for making plywood; that it is resistant to moisture and remains extremely stable at both high and low temperatures, and that it is resistant to scratching and denting. Lowest of the mechanical properties is impact resistance. Laminated paper has a smooth surface requiring no special finish, and indications are that it is not corroded by salt water and that it does not splinter, tear, nor "flower" out when pierced by bullets.

TABLE XVI.—PROPERTIES OF VARIOUS TYPES OF FIBER BUILDING BOARDS.

Board Designation	Specific Gravity	Thickness, in.	Modulus of Rupture, psi.		Nail Holding Strength, lb.	Water Absorption by Volume (2-hr. Immersion), per cent	Water Permeability (Time of Penetration Through Board), hr.
			Across Long Direction	Across Short Direction			
BOARDS MADE FROM CROP PLANT WASTE AND WASTEPAPER[a]							
M	0.252	0.510	397	260	67	6.3	5
AA	0.349	0.530	438	442	112	3.4	6
H	0.410	0.490	831	813	143	4.1	47
BOARDS MADE OF WOOD FIBER[a]							
B	0.260	0.500	396	313	72	12.5	6
O	0.272	0.550	242	227	87	6.8	25
L	0.301	0.510	392	390	71	4.7	16
INTERIOR-FINISH BOARDS WITH SPECIAL SURFACING ON ONE SIDE[a]							
D	0.271	0.480	394	297	79	9.0	18
RR	0.293	0.490	410	351	87	5.6	5
C	0.337	0.470	391	380	72	5.1	26
HIGH DENSITY FIBERBOARD							
	1.020	0.125	6 100				
	1.020	0.250	5 850				
HARD FINISH HIGH DENSITY FIBERBOARD							
	1.110	0.125	10 350				
	1.050	0.250	9 800				

[a] U. S. Dept. of Commerce Report BMS 50, "Stability of Fiber Building Boards as Determined by Accelerated Aging." Tests made under standard atmospheric conditions of 65 per cent relative humidity and 70 F.

Special interest attaches to the development because of its many possibilities.

The properties depend on the paper base used, and processing details, but one type of available commercial paper laminate conforms to the technical specifications in Table XV, put out by an industrial firm.

Heat-Stabilized, Compressed Wood:

Wood is exceedingly compressible in the across-the-grain direction, and it is possible to increase the density of some woods more than twofold and to increase certain properties by such compression. Many commercial adaptations are based on this principle. The main limitation to the process has been the lack of stabilization of dimension in that when subjected to adverse moisture conditions the compressed wood expands greatly and tends to regain its original size. Recent developments have shown that if pressure is applied under certain optimum conditions of temperature and moisture content, compressed wood results which is relatively moisture stable. This process results in the retention of the high shock properties of the wood, and appears very promising for many uses.

Insulation and Building Board:

A great number of insulation and building boards are in common use. This class of boards includes a wide variety of fibrous products ranging from bulky materials weighing from $1\frac{1}{2}$ to 2 lb. per cu. ft. to dense hard boards of more than 60 lb. per cu. ft. The use of this material has expanded very rapidly during the past 15 yr.

These boards are made from both plant and wood fibers. The various species of wood used include spruce, fir, poplar, gum, cottonwood, willow, southern pine, and redwood. In a great many instances these are waste materials of logging, lumbering, or other wood-working industries.

The dense panel boards or hard boards are relatively poor thermal insulators. The grades with greater insulating value are more porous and range from $\frac{1}{4}$ in. to several inches in thickness. The specific weights of these boards range from 14 to 18 lb. per cu. ft.

Generally, the boards consist of two types of fibers: a course fiber to provide structural framework and finer fiber for bonding purposes. Boards of lower density contain a higher percentage of coarse fiber. The better insulating boards generally contain from 30 to 45 per cent fine fiber for binding, the remainder of coarser fiber being from $\frac{1}{2}$ to $\frac{3}{4}$ in. in length. The structural building boards and tiles contain larger amounts of shorter fibers to provide greater density and better finish.

The important physical characteristics of wallboard should include a fair degree of rigidity and water resistance, good flexural and compressive strengths, but low thermal conductivity (Table XVI). Surface texture, hardness, and ability to take paint are also important properties. These products are prepared as standard size wall panels, plank, tiles, lath, roof insulation slabs, and moldings. In building construction they are used as structural building board, for inside and outside sheathing, interior finish, ceiling, flooring, for insulating walls and roofs, and for accoustical treatment purposes.

Varied and extensive use has been found for these products in the war effort, such as sheathing and insulation for Army quarters in cold climates and the protection of supplies. After the war it can be expected that improvement in the properties of boards of this type will bring about wider applications and uses, possibly offering opportunities for further utilization of wood wastes in this field.

"Sandwich" Material:

It is, of course, obvious that in addition to the use of wood species and modified wood and fiber products *per se*, there are practically unlimited possibilities of combining these materials in various forms of composite or "sandwich" construction. In "sandwich" construction, the properties of each material can be used to best advantage, employing where desirable materials of higher density and strength for exterior surfaces where they are most effective in increasing the moment of inertia, as well as in adding to the strength and stiffness of the section. Already well-known is the success attained by this technique in the mosquito bomber, in which plywood faces are glued to a low-density balsa wood core, to afford a lightweight construction of considerable thickness and of relatively high strength and stiffness. Resin-impregnated wood, laminated paper plastic, and many other materials likewise hold promise for innumerable applications in "sandwich" construction.

DESTRUCTIVE AGENCIES AND THEIR CONTROL

> "Little of all we value here
> Wakes on the morn of its hundredth year
> Without both feeling and looking queer.
> In fact, there's nothing that keeps its youth,
> So far as I know, but a tree and truth."
> —*Holmes.*

In considering factually the field of engineering materials, it must be recognized that there is no such thing as absolute permanence under all conditions of service and use. The engineer must be continually on the alert to improve the properties to meet special serviceability requirements and to extend the useful life of the materials. Hence, there is naturally much concern over destructive agencies and their control, and such terms as decay, rust, corrosion, electrolysis, embrittlement, spalling, decomposition, and aging are in everyday use.

Wood is no exception and requires the control of a group of destructive agencies largely peculiar to itself. The principal causes of the deterioration of wood, in their general order of importance, are decay, fire, insects and marine borers, mechanical wear and breakage, weathering and chemical decomposition. Damage or total destruction may result from any one of these causes alone, but it is more common for two or more of them to be at work at the same time.

As a result of research and experience over a great many years, control and remedial measures are available to guard wood effectively against most, if not all, of the destructive agencies to any desired extent. Economic considerations enter the picture also, in establishing a balance between the cost of protective measures, when these costs are substantial, as against replacement. In the problem of control, it is at once essential to know the nature of each of the destructive agencies and the conditions under which it develops.

DECAY

Causes of Decay:

Decay in wood is produced by organisms known as fungi which live on the wood substance. Wood-destroying fungi require favorable conditions for their development with respect to moisture, temperature, and air; hence, if any one of these requirements is eliminated, the growth of fungi will be inhibited. Thus wood that is below a certain critical moisture content will present no decay hazard. Likewise, wood that is maintained at extremely high or low temperatures precludes the development of fungi, just as does the immersion of wood in water to exclude the air.

The one outstanding precaution that must usually be observed in preventing decay in untreated wood is to keep the wood dry, as air-dry wood does not contain enough moisture to permit the growth of wood-destroying fungi. Even the so-called "dry rot" fungi require more moisture than is found in air-dry wood. At moisture contents below the fiber saturation point (25 to 30 per cent) decay is greatly retarded; below 20 per cent, fungus growth is completely inhibited. Fungi grow best and decay is most rapid at moisture contents considerably above 30 per cent.

Keeping wood saturated with water and thus excluding air will also prevent decay. The constantly submerged parts of piling in fresh water, for example, have long life. Likewise, foundation piles under buildings and bridges do not decay if the ground water level is constantly higher than the tops of the piles. Wood buried deeply in the soil, especially if the soil is nonporous, decays very slowly if at all, because air is largely excluded.

Decay Prevention:

For use under conditions favorable to decay, two effective methods of prevention may be employed: (1) select species of high natural decay resistance or (2) resort to preservative treatment. A number of native species of wood have heartwood of high natural decay resistance and offer a means of providing decay protection. It should be pointed out, however, that the sapwood of all species is low in decay resistance. In general, the decay-resistant species are those of which the heartwood contains an appreciable amount of toxic extractives that act as a natural preservative and thus provide decay resistance.

For many outdoor uses, especially those in contact with the ground, the life of untreated wood is too short and preservative treatment must be resorted to for the sake of ultimate economy. The literature concerning the many preservatives offered for use or the numerous methods of applying them is extensive. A background of authentic information on the use and treatment of wood exposed to decay is essential to avoid some of the pitfalls and some of the misinformation on this subject.

One fact of basic importance is that long experience under actual use conditions is required before the effectiveness of any preservative may be considered fully established or can be compared with that of other preservatives, as no laboratory short cut has been found that will give a dependable measure of the effectiveness, reliability, and economy of new preservatives. Long service records are hence invaluable in establishing the effectiveness of various treating agents.

In their respective classes, coal-tar creosote and zinc chloride are the best known and most commonly used preservatives, and each has a hundred years of use to establish the extent of its reliability. Coal-tar creosote is recognized throughout the world as the most effective preservative to use in any situation where the treated wood is exposed to the leaching effect of water. Although creosote is not immune to leaching, the loss of preservative is so slow that a large percentage of the original amount injected into the wood may still remain after 30 to 50 yr. of exposure to water or contact with the soil.

Excellent substitutes for coal-tar creosote are the mixtures of creosote with tar or petroleum which are now so widely used in the treatment of ties and other railway material. The relative cheapness of these mixtures and the fact that they tend to reduce checking and mechanical wear more than offset the increased difficulty of securing good penetration and the greater oiliness of the treated wood, and make them very suitable for railway use. They are not so suitable as straight creosote, however, for poles, piling, and miscellaneous construction timber.

An appreciable number of creosote oils and carbolineums is sold under proprietary brands. As a group, they may be considered as having about the same general properties and degree of effectiveness as common coal-tar creosote. They usually have had some of the ingredients removed that crystallize at low temperatures, so that the preservative remains fluid at much lower temperatures than the creosote generally used for the pressure treatment of ties and timbers. This is definitely a convenience in handling and using the oils.

Zinc chloride is soluble in water and is not nearly so permanent as creosote in the presence of water. Nevertheless, zinc chloride was used in the treatment of railway ties for many years and made them last anywhere from 8 to 16 yr., or even longer, depending on climatic conditions and the thoroughness of the treatment. The superior performance and greater ultimate economy of creosote and creosote mixtures for ties is now generally recognized, and zinc chloride is seldom used for the purpose. As a preservative for building lumber and other material that is not exposed to leaching, however, zinc chloride is still one of the principal preservatives.

A nonproprietary mixture of zinc chloride with $18\frac{1}{2}$ per cent of sodium bichromate has been developed under the name chromated zinc chloride. It is claimed that the addition of the chromium salt very materially increases the resistance to leaching and the effectiveness of the preservative. Such laboratory tests and accelerated field tests as have been reported indicate that chromated zinc chloride is superior to straight zinc chloride, but there has not yet been time for service records to check the results of the small-scale tests.

Mercuric chloride is a very effective preservative with a long record of successful use on a small scale. It is not extensively used because of its high cost, its corrosiveness to iron, and its poisonous character. The oldest commercial wood-preserving plant in the United States, however, began using this preservative in 1848, and at last report was still doing so.

There are numerous other chemical preservatives that are known to have value, including copper sulfate, sodium fluoride, and combinations of copper, zinc, arsenic and chromium salts, as well as a long list of proprietary preservatives. Several of the proprietary salt preservatives are in extensive and successful commercial use and are recognized in specifications.

An important group of special preservatives has been developed for the treatment of millwork. These preservatives consist essentially of a nontoxic, nonaqueous carrier in which is dissolved about 5 per cent of a toxic organic compound. The toxicants most extensively used thus far have been 2-chlororthophenylphenol, tetrachlorphenol, and pentachlorphenol. Copper naphthenate is also being used in similar formulations. The carriers are, for the most part, readily volatile and escape from the wood soon after treatment, leaving only the toxicant behind. In some preservatives, however, the carrier contains a large percentage of nonvolatile, nondrying oils, or certain waxes and drying oils.

Advantages of these preservatives are that they generally do not discolor wood seriously, as does creosote, or change its moisture content, as do the water-borne preservatives.

No discussion of preservatives is complete without some consideration to methods of application. The best of preservatives can do little good if improperly applied. The most dependable treatment is to inject the preservative into wood in closed cylinders by the application of pressure. When correctly done, pressure treatment results in substantial absorptions and deep penetrations, which give a high degree of protection. Careless treatment, however, even by a pressure method, can easily fall very far short of giving the protection that should be obtained.

Hot-and-cold-bath treatment may sometimes be used as a substitute for pressure treatment, but it is more difficult to insure good results or to control absorption and penetration without the use of pressure. Still less effective are brushing, spraying, dipping, soaking, or steeping methods, although they may sometimes be used to advantage when it is impractical to employ more effective methods.

Some preservatives of the type developed for millwork are being promoted for general use by brush or spray application, but it must be recognized that superficial application does not result in sufficient penetration to afford much protection.

A most important fact to remember about all treatments is the necessity of avoiding cutting the wood after treatment. The wood should be cut to final dimensions before treatment wherever this is possible, because cutting after treatment exposes the untreated interior of the lumber to decay. When cutting after treatment is necessary, the cut surfaces should be brushed generously and repeatedly with creosote or with the preservative used in the original treatment.

PROTECTION FROM INSECTS AND MARINE BORERS

The precautions that are effective against decay are, for the most part, effective also against insects. Preservative treatment, for example, protects against both insects and decay. In protection without treatment, however, special precautions are often necessary against insects.

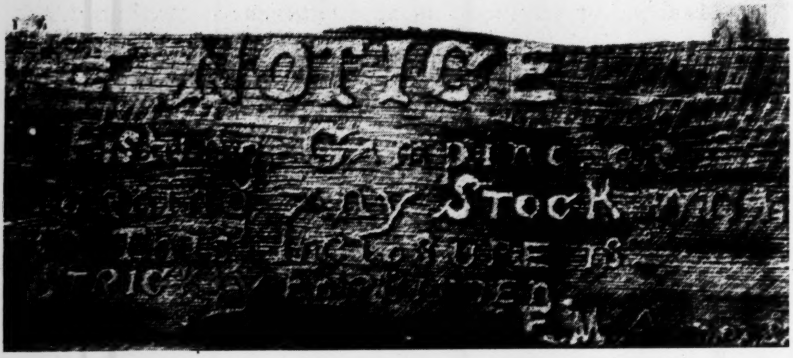
In termite control, as in decay control, it is of primary importance to keep untreated wood dry, well-ventilated, and removed from contact with the ground. All loose wood should be removed from the premises to eliminate the food supplies that would encourage termite visitation. Where additional measures are required, mechanical barriers may be provided to keep the termites from entering the structure. These usually take the form of metal shields that extend over the top of the foundation, beneath the sills, and outward and downward at an angle of 45 deg. beyond the face of the foundation on both sides. Soil poisoning and treatment of the timber in place are also resorted to at times.

Lyctus powder-post beetles confine their activities mainly to the seasoned sapwood of lumber, furniture, flooring, interior trim, tool handles, and similar products. They seldom attack products that are constantly handled and moved about, but work mainly in material that is left undisturbed. Preventing their attack on the woodwork in houses is best accomplished by insuring that the lumber used in the house is sound and free from attack. For stored material, long storage should be avoided and inspections should be made from time to time, so that any attack will be detected before it has made much progress.

When Lyctus beetles are found in wood, all the pieces that are seriously damaged should be removed and burned. The remainder of the material can be treated with toxic chemicals, such as orthodichlorbenzene, or fumigated with suitable materials in a closed container, or heated in a kiln, according to the nature of the product. Strict sanitation and removal of all waste wood that may serve to harbor the insects is important.

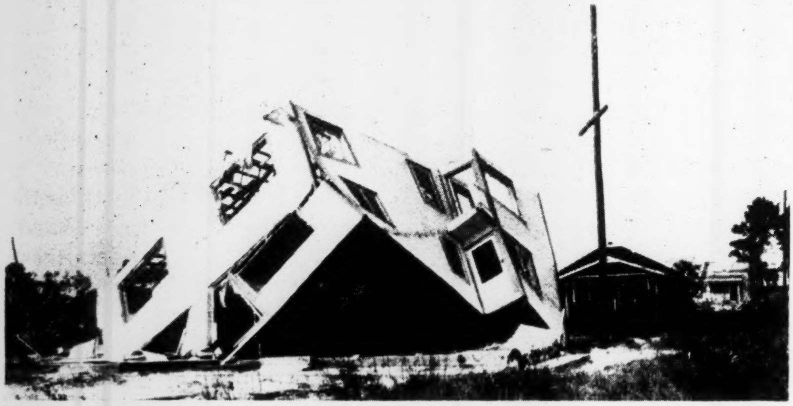
Protection to uninfested wood can be provided by coating all surfaces with paint, varnish, linseed oil, or any other finishing material that fills the pores of the wood and prevents the adult beetles from depositing their eggs in it.

Protection against marine borers in waters where they are plentiful is both difficult and expensive. The most generally useful and practical protection is obtained by treating the piling with maximum absorptions of coal-tar creosote. Nothing less than the maximum amount of oil that can be injected into the wood should be used except in waters where the rate of attack is slow. Heavy creosote treatment is very effective


NOTICE
Camping
Stock
Enclosure is
Strictly Forbidden

The Effect of Weathering May Be Observed on This Signboard, Which Has Been Exposed Many Years Without Painting.

The wood is sound and contains no decay.



This Picture Illustrates Dramatically What Happens to a Poorly Constructed House in a Strong Wind.

Poorly constructed buildings, regardless of material used, are the principal source of loss in storm areas.

A Well-Constructed House That Withstood Two Hurricanes.

Good construction gives an excellent account of itself in hurricane areas.

against borers of the shipworm type, but *Limnoria*, *Chelura*, and *Sphaeroma* have been known to work even in creosoted wood. Depth of penetration, as well as high absorption, of the creosote is highly important, for the borers quickly detect any weaknesses in the treatment and get through to the untreated wood in the interior, where they can work unhindered.

Numerous attempts have been made at various times to prevent destruction of piling by marine borers through the application of special paints, or combinations of paint, wood battens, and fabrics, but with very little success. Covering the piles with sheet copper or copper alloys has been found somewhat more effective than the paint methods, but it is not a reliable protection. A thick coating of gunite very carefully applied (at about twice the cost of creosoting) was used for the protection of 1400 Douglas fir piles in the harbor of Tacoma, Wash. After 14 yr. service, the piles were reported to be free from borer attack. Still more expensive, but also probably more effective, is the method of surrounding creosoted piling with precast concrete cylinders, or cast iron pipe sections.

PROTECTION FROM FIRE, WEAR, AND CHEMICAL DETERIORATION

Fire resistance can be imparted to wood by impregnating it with substantial quantities of suitable chemicals, notably the phosphates of ammonia, or mixtures of ammonium phosphate, ammonium sulfate, borax, and boric acid. The use of "fireproofed" wood under peace-time conditions was generally limited to places where it was required by building codes and a few other places where high resistance to fire was considered of maximum importance. At the present time, large quantities of "fireproofed" wood are being used in the construction of "lighter-than-air" hangars, and other structures necessitated by the war. Some degree of fire resistance can be provided by fire-resistant coatings, but unfortunately, despite the best efforts of investigators, effective coatings that are practical and that retain their effectiveness under exposure to the weather over a long period are lacking. For interior use, there are several very efficient coatings.

An effective way to provide fire safety in wood structures is to design them to provide slow burning construction, thus making it possible to confine fires to limited portions of structures, to prevent rapid spread of flames, and to facilitate the safe and prompt escape of the occupants. Fire stops in walls, for example, delay the spread of fire within the walls and greatly increase the opportunities to detect and extinguish the fire before it is out of control. Industrial structures properly designed and built with large timbers are slow burning and may be safer from fire than some buildings built of so-called "incombustible" materials. The contents of the building are often more important from the fire standpoint than the materials of construction, for inflammable contents can burn in any building.

Wood is naturally resistant to rapid destruction by contact with most chemicals, but is destroyed in time by strong acids and alkalies and certain other materials. Impregnation of the wood with paraffin or similar materials increases its resistance to chemical action to some extent, but has only limited effectiveness. Impregnating the wood with a solution of resin-forming materials and then bringing about the resin-forming reaction within the wood offers promise of increased chemical resistance. No practical protection has yet been found against the deterioration of wood continuously exposed to heat.

Weathering, as a destroyer of wood, can usually be neglected except where the appearance of weathered wood is objectionable. In these uses, weathering is prevented by keeping the wood painted.

EFFECT OF TIME OR LENGTH OF SERVICE ON THE STRENGTH OF WOOD

It has been shown that wood, like other materials, is subject to attack by various destructive agents but that methods are in general available to prolong its life so that various practical service requirements can be met under conditions where destructive agents are encountered. There is ample historical evidence of its permanence when suitable protection is provided.

So far as is known, the lignin and cellulose which constitute the wood substance are not subject to chemical changes with time when kept dry, although the color of wood may be slightly changed by long-continued exposure to air. Possibly this change of color results from oxidation of substances that are not parts of the wood substance.

A recent survey has shown that literally hundreds of well-designed bridges made entirely or partly of wood have served satisfactorily and with but little attention for long periods. Many that are more than a century old are still in service. Others, while still in satisfactory condition, have been altered or replaced to meet the demands for greater width of roadway and higher load capacity than obtained when they were built.

In Europe, many wood structures or structural units that are centuries old are still in existence. Perhaps the most notable are the trusses in the Basilica of St. Paul at Rome, part of which was constructed in 816, during the Pontificate of Leo III. Certain such roof trusses are known to have given service for over 1000 yr.

> 24. Therefore whosoever hereth these sayings of mine, and doeth them, I will liken him unto a wise man, which built his house upon a rock:
>
> 25. And the rain descended, and the floods came, and the winds blew, and beat upon that house; and it fell not, for it was founded upon a rock.
>
> —St. Matthew.

THE FUTURE OF WOOD

> "My gran'ther's rule was safer'n 't is to crow:
> Don't never prophesy—onless ye know."
>
> —*Lowell.*

Though wood and wood products have a long tradition as important structural and engineering materials, during the past decade an awakened interest in their use and application has been stimulated by significant research developments and more recently by the exigencies of war. Contributing to this renaissance of wood have been marked advancements in at least several broad lines, as follows:

1. Improvement in joints and fastenings, usually the critical feature of structural timber design.
2. Better structural grades, affording more precise strength evaluation and more uniform quality.
3. Improvements in glue and gluing technique, including the development of synthetic resins that afford essentially waterproof joints.

4. The development of laminated arch construction, including establishment of design data and fabrication techniques.

5. Extensive developments in plywood production, making available a large variety of constructions, with moisture-resistant properties if desired.

6. Advent of chemical seasoning methods, which not only result in quicker drying, but in the practical elimination of drying defects.

7. Increased facilities for wood preservation, affording opportunity for increased life and serviceability and reduced maintenance costs.

8. Composite constructions, using wood with other materials, and employing to best advantage the salient properties of each.

9. Improved wood, plastics, and paper-base laminates, involving chemical conversion or resin impregnation, offering a variety of products that are finding many new uses.

10. A wide variety of fiberboards with properties designed to meet particular requirements.

11. New developments in molding and laminating techniques, permitting the bending, forming, and assembly of complicated and intricate structural parts, with either laminated or plywood constructions.

It has been strikingly obvious that most of the engineering handbook data on wood have become more or less out of date and generally inadequate. The preparation of the Wood Handbook by the Forest Products Laboratory presented the first comprehensive, correlated attempt to bring together under one cover the most recent basic data and related information on wood. Issued in 1935, this publication has had wide circulation, but is already in need of extended revision. When publication restrictions are removed after the war, a wealth of new data will be available for release that will facilitate design in wood, plywood, and the great variety of new and composite products. The publication of current data, together with the significant development along the lines indicated, is already having an effect in the modernizing of handbook data and should provide also a basis for more adequate consideration in the curricula in engineering education.

Experience with wood during the war in an almost unlimited variety of uses has shown that in its many forms it is one of the most versatile of materials at the command of the engineer. Fabrication facilities have already undergone a marked increase, and the fabrication of various manufactured products, such as structural units of various kinds, prefabricated houses, and a great variety of other products, can be expected.

There seems but little doubt also that post-war development will bring new combinations of the older established materials and the variety of new products yet to come, now under development, or already on the market, to afford constructions and uses that effectively take advantage of the best qualities and properties of each for a given purpose. Marked progress and success already made in bonding wood to metal, for example, presage many applications and unique uses.

And then, above all, research must continue to carry the burden of providing the needed data, new developments, and new conceptions that will make wood and its host of related products even more than ever before the versatile servant of man in the more complex and changing modern world.

What of future forest supplies? Nature is both a strict taskmaster and a generous producer. Trees are a crop that, with even scant encouragement, is ready to grow and reproduce in kind. Perpetual crops can be ours by merely learning to cut timber selectively, so that only trees of a desirable size are cut, or by following other approved forestry practices, to the end that continuing production is maintained for permanent industries and stable communities.

What of future forest supplies? Says Lyle F. Watts, Chief, Forest Service, United States Department of Agriculture:

"Wood, in contrast to so many other engineering materials, is a crop and the supply can be renewed indefinitely. But like other crops produced from the soil, the growing of the needed quantity and quality of product requires the application of sound management practices. Except where the stands consist chiefly of virgin timber, as in parts of the West, the quantity of timber cut each year should not exceed the growth of usable timber. The only acceptable philosophy for balancing the drain with the growth is to grow more timber—not to use less. If reasonably good forest practice is applied generally throughout the country we may expect to build up forest growing stock in the course of the next century to yield a cut of 21 billion cubic feet annually. This is substantially in excess of the current rate of utilization and should be ample for all foreseeable needs."

"Man of the Future, what shall be
The life of Earth that you shall see?
What strange new facts the years will show?
What wonders rare your eyes shall know?
To what new realms of marvel, say,
Will conquering science war its way?"
—*Bennett.*

THE MEASUREMENT OF A-C. AND D-C. PERMEABILITY ON 28-CM. TEST SPECIMENS

By J. P. Barton[1] and G. W. Smith[1]

Synopsis

The Tentative Method of Test for Permeability and Core Loss of Flat-Rolled Magnetic Materials Using 28-cm. Specimen (A 257 - 42 T)[2] provides a means for measuring a-c. permeability of flat-rolled magnetic materials using a double lapped joint core assembly. Investigation to date indicates that the same test frame and core assembly can likewise be used for making d-c. permeability measurements.

Typical measurements of a-c. and d-c. permeability are shown, made on the same core assembly, of test specimens of material having a large range in permeability and silicon content with good agreement being obtained. Comparison is also made with data obtained using a Fahy permeameter. The skin effect due to frequency in reducing the permeability in the vicinity of maximum permeability is also shown, the magnitude of this effect being a function of resistivity, thickness, frequency, and the d-c. permeability.

It is felt that if further investigation of d-c. tests for permeability by this method are judged satisfactory, several of the current A.S.T.M. standard methods of test for core loss, permeability, hysteresis, etc., can be combined into one, using only one test frame and specimen assembly for a-c. and d-c. tests.

In the measurement of the magnetic properties of flat-rolled materials, it is desirable to obtain all the data on one specimen size and core assembly so that the information can be definitely correlated. This paper reports the results of tests on a-c. and d-c. permeability and indicates a way toward realization of this goal.

During the round-robin correlation work, leading to the writing of A.S.T.M. Tentative Method of Test for Permeability and Core Loss of Flat-Rolled Magnetic Materials Using 28-cm. Specimen (A 257 - 42 T),[2] it was understood that several laboratories[3] had used the double-lapped joint core assembly of 3 by 28-cm., or longer, strips for d-c. permeability tests. The question arose as to the possibility of making all magnetic tests on specimens arranged in what may be called a basic four-sided core assembly, composed of 3 by 28-cm., or longer, strips using a double-lapped joint. Our laboratory undertook comparison tests of the a-c. and d-c. permeability and after a series of tests of many specimens, we report herein the

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[1] Manager, Bureau of Electrical Sheet Sales, and Magnetic Testing Engineer, Vandergrift Works, respectively, Carnegie-Illinois Steel Corp., Pittsburgh, Pa.

[2] 1942 Book of A.S.T.M. Standards, Part I, p. 1091.

[3] Westinghouse Research Laboratories, Westinghouse Electric and Manufacturing Co., and Bell Telephone Laboratories, Inc.

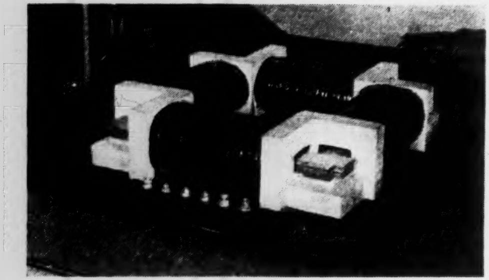

FIG. 1.—Test Frame, Showing Specimen in Place.

results of four representative tests on materials having a wide range of permeability.

The a-c. permeability test method followed the Tentative Method A 257 except that requirements for the number of turns of the windings differed. This tentative method is based on, and represents the outgrowth of, the work of S. L. Burgwin,[4,5] and others. The d-c. permeability test method used the same frame and specimen setting as for the a-c. test, with the frame being substituted for the Fahy permeameter, but using the Fahy control equipment.

The test frame is shown in Fig. 1. It has the following dimensions:

Bakelite tube, length = 19.7 cm. ($7\frac{3}{4}$ in.)
Inside diameter = 1.75 by 3.65 cm. ($\frac{11}{16}$ by $1\frac{7}{16}$ in.)
Wall thickness = 0.16 cm. ($\frac{1}{16}$ in.)
Winding length = 18.4 cm. ($7\frac{1}{4}$ in.)

FIG. 2.—Apparatus Used for a-c. Tests.

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[4] S. L. Burgwin, "A Method of Magnetic Testing for Sheet Material," *Review of Scientific Instruments*, Vol. 7, July, 1936, pp. 272–277.

S. L. Burgwin, "Measurement of Core Loss and a.c. Permeability with the 25-cm. Epstein Frame," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 779 (1941).

[5] About 1932, S. L. Burgwin, of the Westinghouse Electric and Manufacturing Co., was faced with a problem of measuring the core loss and permeability of four strips, 3 by 25 cm. The solution seemed best satisfied by arranging the strips in the form of a square with the ends completely overlapping, thus forming a low-reluctance joint and a closed magnetic circuit composed of all the test materials. Further analyses and tests, using a greater number of strips (in multiples of 4), gave good results leading to both a-c. and d-c. tests during the following years. Later, J. A. Ashworth of the Bell Telephone Laboratories, Inc., investigated this method and found it of preferred value in magnetic measurements, especially for materials of high permeability, as did Mr. Burgwin.

Winding per solenoid:
- Winding No. 1 (inner) 265 turns (2 layers) No. 17 silk-covered enameled wire
- Winding No. 2 (middle) 26.5 turns (1 layer) No. 17 silk-covered enameled wire
- Winding No. 3 (outer) 265 turns (7 layers) two No. 13 silk-covered enameled wire in parallel

Total resistance of four solenoids in series:

Winding No. 1	2.385 ohms
Winding No. 2	0.325 ohms
Winding No. 3	0.748 ohms

The test frame of Tentative Method A 257 is recommended, however, with a third winding, located between the two specified windings, to be added at 17.5 turns average per solenoid or a total of 70 turns.

For the a-c. tests, windings Nos. 1 and 3 are used, the former for the potential circuit, and the latter for the magnetizing current. For the d-c. tests, winding No. 3 constitutes the magnetizing winding, with winding No. 1 being used for low flux densities, and winding No. 2 for the higher flux densities. Figure 2 shows the essential equipment (not connected but grouped closely to illustrate) used for the a-c. test, with Fig. 3 showing the essential equipment used for the d-c. tests. The two mutual inductors shown in Fig. 2 are essentially identical to those described in Tentative Method A 257.

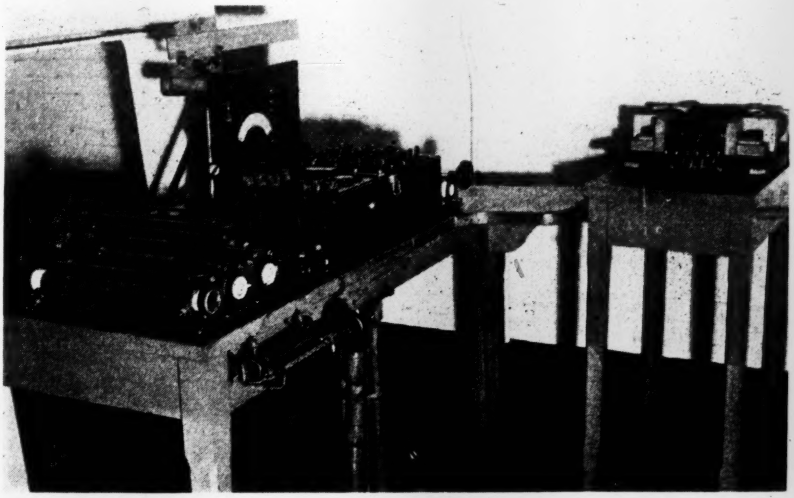

FIG. 3.—Apparatus Used for d-c. Tests.

In making the a-c. tests with this apparatus, the flux voltmeter deflections, used in determining the magnetizing force, in oersteds (H), were too small for satisfactory reading at flux densities below 7 kilogausses, the lowest voltage scale on the meter being 0 to 75 v. In order to obtain values down to 2 kilogausses as well as comparative test data on the same specimens from another laboratory, Burgwin of the Westinghouse Research Laboratories agreed to duplicate our tests, using a full-wave mechanical rectifier in series with a milliammeter in place of a flux voltmeter, which allowed greater sensitivity. The data he obtained substantiated our tests, and in view of this, his values for a-c. permeability were used below about 7

TABLE I.—TEST SPECIMEN DATA.

Specimen	Strip Thickness, in.	Total Number of Strips	Strip Size, cm.	Fahy Test, Strips Used	Total Weight, g.	Cross-Sectional Area, sq. cm.	Silicon Content, per cent	Strip Grain Direction
Fig. 4	0.014	92	3 x 53	10	3792	2.385	3.25	All Length
Fig. 5	0.014	96	3 x 53	10	3564	2.24	4.5	Half and Half
Fig. 6	0.0185	72	3 x 53	10	3779	2.376	3.2	Half and Half
Fig. 7	0.025	56	3 x 53	8	3878	2.376	0.6	Half and Half

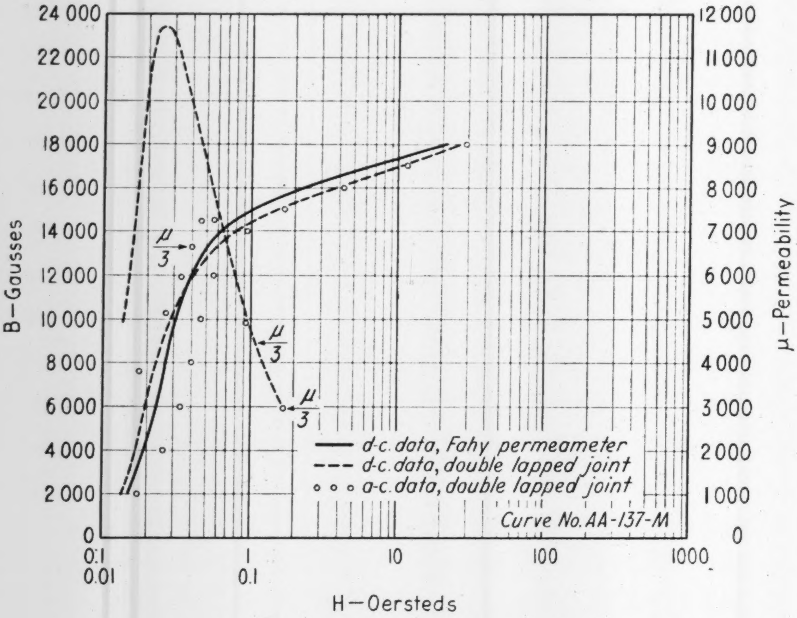

FIG. 4.—Normal Magnetization Curves.

0.014 in. thick, 3¼ per cent silicon steel, cold reduced high-permeability steel, specimen strips 3 by 53 cm., all lengthwise.

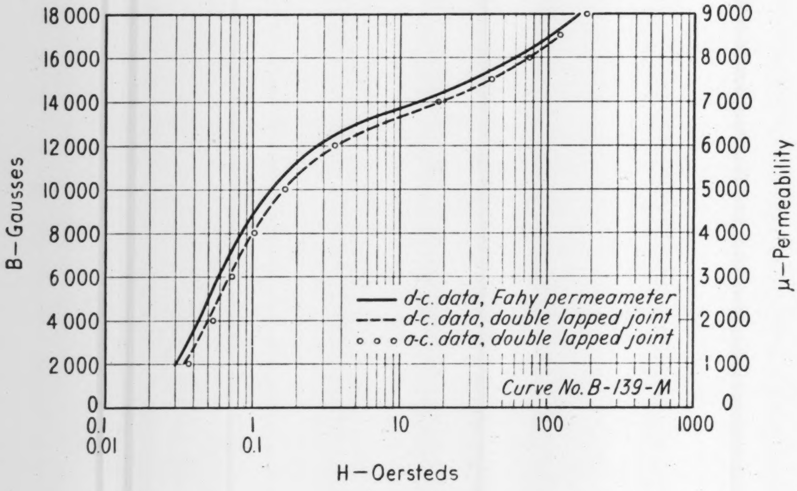

FIG. 5.—Normal Magnetization Curves.

0.014 in. thick, 4½ per cent silicon steel, hot-rolled annealed sheets, specimen strips 3 by 53 cm., half lengthwise, half crosswise.

kilogausses in Figs. 4 to 7. The test specimen data are given in Table I.

Three magnetization curves are shown in each of Figs. 4 to 7. The solid line curve is from data taken on the Fahy permeameter using a portion of the test specimen with the number of strips used given in Table I. The dashed curve shows d-c. magnetization data, and the circles show the a-c. magnetization data, both obtained on the same core assembly of the specimens in the test frame described above, using the double-lapped joints. Specimens 3 by 53 cm. were used as they were part of a group involved in correlating the core loss and permeability between the 50-cm. and 25-cm. test frames, double lap joint and

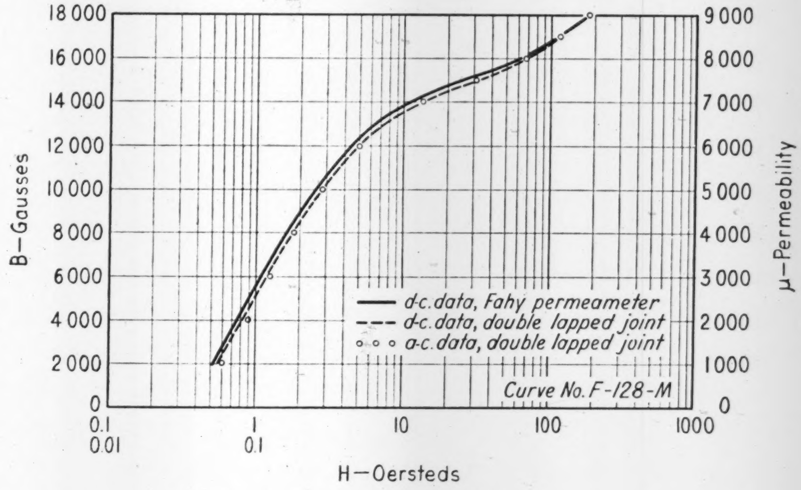

FIG. 6.—Normal Magnetization Curves.

0.0185 in. thick, 3¼ per cent silicon steel, hot-rolled annealed sheets, specimen 3 by 53 cm., half lengthwise, half crosswise.

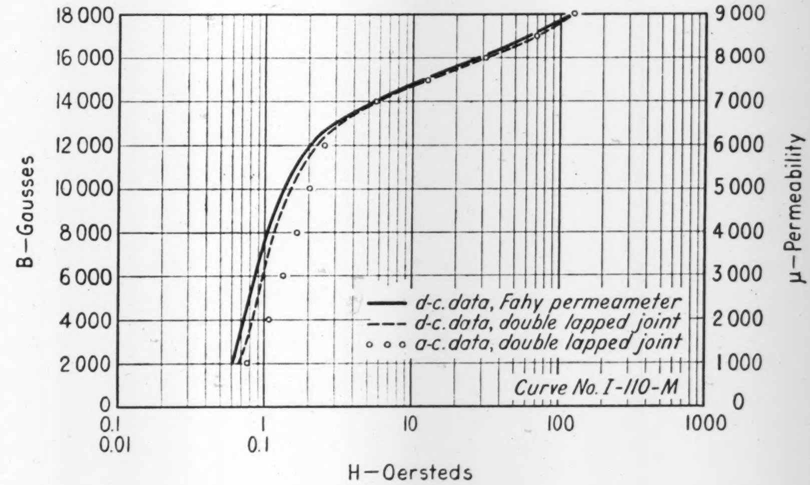

FIG. 7.—Normal Magnetization Curves.

0.025 in. thick, 0.6 per cent silicon steel, hot-rolled annealed sheets, specimen strips 3 by 53 cm., half lengthwise, half crosswise.

butt joint. The effect of the specimen material projecting beyond the corner joints is negligible, but serves to counterbalance the portion of the specimen strips between the corner joints.

With reference to Fig. 4, the magnetic properties of the material involved have a high degree of directionality, with the permeability being high in the direction of rolling when compared with the permeability perpendicular to the direction of rolling. The difference between the d-c. permeability, as measured on the Fahy *versus* the double-lapped joint, appears to be a characteristic difference observed on materials having maximum permeabilities over about 10,000, whether on silicon steel or nickel-iron alloys. This also has been observed by several other laboratories.

Of particular interest in Fig. 4 is the difference between the d-c. and a-c. magnetization double-lapped joint test, below 14 kilogausses, due to the skin effect depressing the d-c. permeability in the vicinity of maximum permeability. The permeability curves are shown in addition to the magnetization curves, in Fig. 4 only, and it will be observed that the maximum permeability is reduced from 35,100 d.c. to 21,900 a.c., at 60 cycles. Tests at 30 cycles show less depression of permeability than at 60 cycles. Also to be observed is the slight depression of the a-c. magnetization curve compared to the d-c. curve above 14 kilogausses, due apparently to the fact that the permeability is still sufficiently high to be affected by the skin effect. This is illustrated by comparing Fig. 4 with Figs. 5 and 6, which cover much lower permeability materials.

The magnitude of the effect of frequency in depressing permeability (skin effect) is a function of the strip thickness, the resistivity, and the permeability of the material. As the frequency is increased, the eddy currents in the test strips cause a nonuniform flux distribution throughout the strip thickness, crowding the flux toward the strip surface.

For a given frequency, the thicker the strip, the greater are the eddy currents; also the lower the resistivity, the greater are the eddy currents. At this point, reference is made to Fig. 7, which shows a reduction of permeability below 14 kilogausses, similar to the reduction illustrated in Fig. 4 but due to the increased thickness and reduced resistivity. This is to be expected, even though the material characteristics differ to a large extent, as stated in Table II.

TABLE II.

Material	Strip Thickness, in.	Silicon Content, per cent	Resistivity, microhms per cu. cm.	Maximum d-c. Permeability
Fig. 4	0.014	3.25	50	35 100
Fig. 5	0.014	4.50	64	8 700
Fig. 7	0.025	0.60	20	7 000

Reference is also made to Fig. 5 which shows essentially no depression of maximum permeability due to skin effect, even though the strip thickness is the same as Fig. 4 and the silicon content is higher. The lack of depression is essentially due to the permeability being only 24 per cent of that for Fig. 4. Similarly, even though the permeability of Fig. 7 is only 20 per cent of Fig. 4, the large increase in strip thickness and decrease in resistivity are sufficient to depress the permeability to quite an extent.

Although this phenomenon is well known, current test equipment precludes readily measuring it. Practically all published magnetization data are based on d-c. tests, although the great majority of silicon steel is used on cores subject to 60 cycles. Therefore, test means to show readily the effect of frequency on magnetization curves when compared to the d-c. curves on the same test specimen is of importance to designers.

Referring to Figs. 5 and 6, it will be observed that good agreement exists between the a-c. and d-c. tests. Figures 5 and 6 show a small depression of maximum permeability due to frequency. As illustrated in Figs. 5, 6, and 7, the Fahy curve is displaced to the left, and this has been consistently observed on all specimens measured to date, of various thickness and silicon content and having maximum permeabilities under about 10,000.

There seems but little doubt that the double-lapped joint test method can be readily extended to flux densities lower than 2 kilogausses, especially for the d-c. tests. Below 1 kilogauss, however, the low-induction a-c. bridge test method, A.S.T.M. Standard Methods of Test for Magnetic Properties of Iron and Steel (A 34 – 42)[6] is of more value and better suited.

No technical disadvantages of any consequence have been encountered to date, but the advantages of the double-lapped joint test method are many. First, the entire magnetic circuit is composed of the material under test, which is the ultimate goal in all magnetic testing. The magnetic circuit is closed with the joint reluctance very low and the joint-flux density at half value of that for the core between the joints. The test windings are uniformly distributed over a large percentage of the mean length of the magnetic circuit which in turn is of uniform cross-section throughout, except for the joints. The specimen strips can be cut and tested at any angle to the rolling direction, or a composite specimen assembled from strips cut in various ways. This method is of particular value for materials having maximum permeabilities at least up to 50,000, and having high permeabilities at flux densities over 12 kilogausses, such as cold-reduced silicon having highly directional magnetic properties.

The same test specimen core assembly can be consecutively tested for d-c. and a-c. permeability, hysteresis loops, core loss, and incremental permeability and core loss, thus allowing definite correlation of all these tests. The weight of the specimen can be varied over a wide range, from a minimum of four strips to as many as desired, in multiples of four, depending upon the sensitivity of the measuring instrument and the windings of the test frame. Furthermore, a standard simple frame easily constructed is substituted for a permeameter, such as the Burrows or Fahy, with the balance of the apparatus needed being usually readily available in most electrical testing laboratories. It was found that the speed in testing is improved under some conditions, using the frame and method reported herein.

CONCLUSIONS

In conclusion, it may be stated that from the evidence observed to date, including the representative test data reported herein, the double-lapped joint core assembly offers an improved, useful, and flexible means for measuring a-c. and d-c. permeability in the flux density range of at least 2 to 18 kilogausses. As the core loss and a-c. permeability test is covered by Tentative Method A 257,[2] the incremental permeability and core loss by A.S.T.M. Tentative Method of Test for Incremental Permeability and Core Loss of Flat-Rolled Magnetic Materials at Low Alternating Inductions Using 28-cm. Specimen (A 258 – 42 T),[7] and the d-c. permeability test results reported to date by several laboratories, are regarded as satisfactory, then the same core assembly can be undoubtedly used for hysteresis loop data. Subcommittee III on Direct Current Test Methods of the Society's Committee A-6 on Magnetic Properties is currently investigating the final acceptability of the double-lapped joint core assembly method for d-c. magnetization testing. If its findings substantiate prior work and the method is approved, then we can look forward eventually to consolidating all these test methods, using one specimen core assembly and test frame.

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[6] 1942 Book of A.S.T.M. Standards, Part I, p. 651.

[7] 1942 Book of A.S.T.M. Standards, Part I, p. 1097.

DISCUSSION

MR. T. SPOONER.[1]—I should like to take this opportunity to thank the Carnegie-Illinois Steel Corp. for the very considerable contributions which it has made in the past few years to magnetic testing.

The paper by Messrs. Barton and Smith should be considered in connection with the paper which was presented by Mr. Burgwin before this Society two years ago.[2] It is supplementary to it, or, perhaps, I should say complementary. Barton and Smith have taken the method originally proposed by Burgwin and put it in a commercial form. They have shown that it is a very sound and useful method.

When skin effect is negligible, as the curves in the paper show, the checks are remarkably close between the a-c. and d-c. tests. I was very much astonished when I first saw these results to see how closely they did agree. We have then a very simple permeameter suitable for a-c. and d-c. tests and one which I think is going to come into very common use. In fact, this is so simple that it is just as easy to operate as the old Rowland ring method for testing ring samples. When we think of the complications and the difficulties in operating the Burrough's permeameter we can appreciate what a great advantage this is, particularly in obtaining hysterisis data. It should be remembered, however, that this method is only applicable to laminated materials and to moderate inductions. It will be necessary to use the standard permeameters for very high inductions and particularly for permanent magnet materials.

The possibilities of making a-c. and d-c. permeability tests so simply and accurately are very attractive, particularly in connection with the new orientated materials having very high permeability. For these materials permeability is just as important as core loss, and the possibility of making these two tests simultaneously are going to be greatly appreciated by the men who have to test these materials.

I believe also that the possibility of making a-c. permeability tests so easily will result in the adoption of the effective a-c. values rather than d-c. values for laminated materials. There is another reason for this—most laminated materials are used under a-c. conditions and we are really more interested in effective values than we are in actual d-c. values.

Messrs. Barton and Smith have made a very considerable contribution to the art of magnetic testing, and I am sure that we are going to appreciate in the future even more than we do now what this contribution means.

MR. G. SHOMBERT, JR.[3]—The method of measuring a-c. permeability is based, of course, on the use of a rectifying type voltmeter for the determination of both B and H. For measuring B, the flux voltmeter is entirely satisfactory. The

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[1] Manager, Engineering Laboratories and Standards Dept., Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] S. L. Burgwin, "Measurement of Core Loss and a.c. Permeability with the 25-cm. Epstein Frame," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 779 (1941).

[3] Assistant Superintendent, Test Dept., Allis-Chalmers Manufacturing Co., Pittsburgh Works, Pittsburgh, Pa.

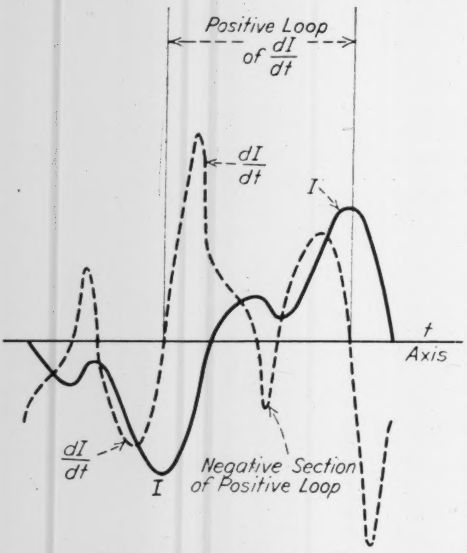

FIG. 1.—Exciting Current Wave, I, and Its Derivative, $\frac{dI}{dt}$.

value of H is obtained by measuring the IX drop across an air-core inductor; Camilli[4] has shown that

$$e = L\frac{dI}{dt}$$

$$\text{and hence } e_{\text{avg.}} = kLI_c$$

where I_c is the "crest value" of the current and is proportional to $H_{max.}$ Now if the exciting current wave has more than one peak per half cycle (Fig. 1), the derivative will have a negative section in the positive loop, and *vice versa.* Under this condition, the rectifying-type voltmeter is inaccurate, for with a half-wave rectifier the negative section is ignored, or with a full-wave rectifier it is turned above the line and added instead of subtracted. In either case a false result is obtained.

While this type of current wave may not be encountered very often, the possibility of its occurrence must be kept in mind whenever tests of this nature are conducted.

MR. S. L. BURGWIN.[5]—As far as I know I have never seen a curve like that shown by Mr. Shombert in the tests I have made. In other words, while the point Mr. Shombert has made is theoretically correct, I do not think such conditions would ever arise in the 25-cm. test specified by the A.S.T.M.

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[4] G. Camilli, "Reduction of Transformer Exciting Current to Sine-Wave Basis," *Journal*, Am. Inst. Electrical Engrs., Vol. 46, May, 1927, p. 692.

[5] Research Engineer, Industrial Control Engineering, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

A TEST FOR MEASURING DRAWABILITY OF DEEP-DRAWING STEELS

By Francis W. Boulger[1] and F. B. Dahle[1]

Synopsis

The reasons for the failure of present standard tests to evaluate the suitability of sheet metals for deep-drawing operations are discussed and an experimental testing method is proposed. This method utilizes a cup-drawing procedure which integrates the properties in a steel needed to satisfy the drastic drawing operations met in service.

The data indicate that the cup-drawing test method is accurate and reproducible in showing differences between materials of ostensibly similar quality. A comparison of a few test results with service performance figures shows a good correlation between press breakage and cup-drawing ratings. The behavior in the experimental test equipment rated materials in a different order than tension, hardness, and cupping tests. This does not disprove the suitability of the experimental method because the standard methods are known to be unsatisfactory. The data indicate that increases in carbon and sulfur contents lower the cup-drawing ratings but that variations in manganese, copper, and phosphorus, for the ranges covered by the sample group, were without effect. The results show that temper rolling lowers the cup-drawing ratings and that the effect is most pronounced in the neighborhood of 0.75 per cent reduction.

No mechanical test in use at present gives an entirely adequate or reliable indication of the behavior to be expected from sheet metal used in industrial deep-drawing operations. This fact has been discussed by Gillett (**1**),[2] Asimow and Crombie (**2**), Jevons (**3**), and many others. The failure of the standard testing methods to predict success in drawing applications is emphasized by the present practice of buying sheet steels on the basis of performance rather than by specifications. The situation is regrettable because the application of tests constitutes the only means of checking the quality and uniformity of the materials passing from supplier to fabricator.

Deep-drawing operations on sheet metal require the absorption and distribution of the applied forces by definite amounts of deformation and stress. The properties of the material which affect its behavior under such conditions are chiefly its rupture strength, its rate of strain hardening, and its ability to flow under the conditions imposed. The determination of these fundamental properties and their proper evaluation should make possible reliable predictions of service performance. When the service conditions are as complex as they are in deep drawing, however, simulative tests usually offer better means of forecasting service behavior.

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[1] Research Metallurgist, and Assistant Supervisor, respectively, Battelle Memorial Institute, Columbus, Ohio.

[2] The boldface numbers in parentheses refer to the papers appearing in the list of references appended to this paper, see p. 517.

In deep drawing sheet metal, the principal stresses resulting in the fabrication of the desired form are bending, stretching, and upsetting actions. All of these forming stresses have been simulated more or less by various tests employed for measuring the quality of sheet materials. Bending actions ordinarily present no problem although they have been the subject of some nonstandard tests (4, 5). Stretching always accompanies drawing operations, and when a metal fails in drawing, it almost invariably fractures in a region subject to stretching. This fact has led to the popular belief that the most satisfactory materials for deep drawing are those which show the maximum amount of elongation without fracture. Therefore, considerable emphasis, in classifying deep-drawing materials, has been placed on elongation figures from tension tests and cupping values from tests of the Olsen or Erichsen type.

Both the tension test and cupping tests may be capable of differentiating between good and bad materials for deep drawing because they show the behavior in pure stretching action. Their failure to make the close distinctions necessary in exacting applications is because of limitations inherent in the methods. By their neglect of the other drawing stresses, the methods fail to reproduce drawing conditions closely enough to distinguish between materials which have only slight or critical differences in drawability.

The loading conditions encountered in deep drawing are at least biaxial and possibly triaxial; whereas, the tension test provides information on behavior under uniaxial stress. The ordinary cupping test shows the behavior under simple stretching action. During deep-drawing operations, the stretching action is largely determined by the conditions in other regions. It is controlled by the resistance to upsetting and by the strain hardening characteristics of the material. The usual cupping tests are helpful, however, because they give an indication of directional differences and grain size variations of materials and suggest the probable surface finish of drawn articles.

One nonstandard test which has shown considerable promise in detecting differences in the drawability of materials is the so-called "wedge-draw" test developed by Sachs (6) and others. Recently the method has been refined by Brewer and Rockwell (7) to evaluate the drawing qualities of aluminum alloys. The wedge-draw test reproduces the conditions which occur in a segment of a blank when it is being drawn into a cup. Since the segment is wedge shaped, the test simulates the combined tensile and compressive stresses that exist in drawing operations by pulling a wedge-shaped specimen through a die. The method neglects bending stresses normally present in drawing operations but is said to give a good correlation with service performance. The wedge-draw method provides a tool for studying the stress-strain relationships in deep-drawing but is not particularly suited for routine testing of materials of closely similar properties.

With these factors in mind, the ordinary cupping test was modified to include bending and upsetting actions in the testing operation. The adaptation resulted in setting up a single stage deep-drawing operation for studying sheet materials. By this method the materials are classified on the basis of their drawing performance under fixed conditions. In contrast to the wedge-draw test, it provides an actual rather than a simulated drawing operation. It gives a closer differentiation between materials, because each blank is made up of a multitude of tiny wedges, and the success or failure of the cup-drawing operation is the integrated result of their behavior.

A satisfactory test for evaluating the drawing quality of sheet materials must fulfill several requirements. The most important of these is that it must rate the materials in the same order as actual performance in the presses. Presumably a reliable test will rate materials in a slightly different order than the usual tension, hardness, and cupping tests because these are known to be not completely satisfactory. An adequate testing method must be consistent, reproducible, and differentiate between materials of only slightly different quality. It is desirable, but not essential, that the test method be simple and rapid in application. It is the purpose of this paper to describe an experimental cup-drawing test and present some data showing how it meets the criteria for a helpful source of information in studying and evaluating drawing quality.

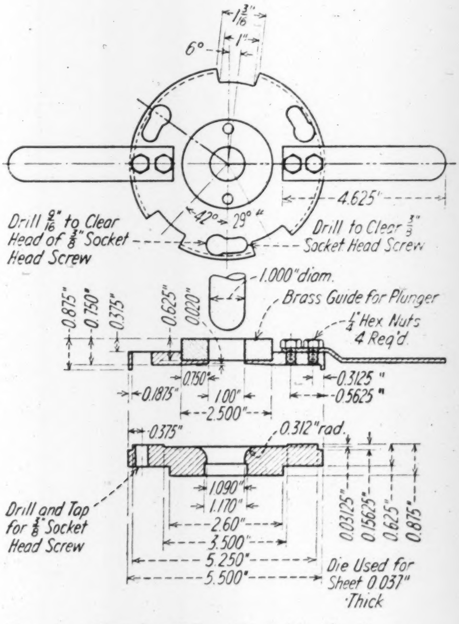
1 3/16"
1"
6°
4.625"
42°
29°
Drill 9/16" to Clear Head of 3/8" Socket Head Screw
Drill to Clear 3/8" Socket Head Screw
1.000" diam.
Brass Guide for Plunger
1/4" Hex Nuts 4 Req'd.
0.875"
0.750"
0.375"
0.625"
0.020"
0.750"
0.1875"
1.00"
2.500"
0.3125"
0.5625"
0.375"
0.312" rad.
1.090"
1.170"
2.60"
3.500"
5.250"
5.500"
0.03125"
0.15625"
0.625"
0.875"
Drill and Tap for 3/8" Socket Head Screw
Die Used for Sheet 0.037" Thick

FIG. 1.—Sketch of Cup-Drawing Equipment.

CUP-DRAWING TEST METHOD

The testing method consists of drawing flat, circular blanks into round-bottomed cylindrical shells by means of a simple die and plunger. The inside diameter of the cups formed is 1 in., and the heights vary with the size of the blanks tested. The index of the drawing quality of a material in this test is considered to be the maximum diameter of blank which can be formed and the percentage breakage in that size. Ten disks of the critical size are tested, and the results are used as the basis of comparison. Data are also recorded for maximum diameter of blanks drawn with no breakage, and diameter of blanks which all fail.

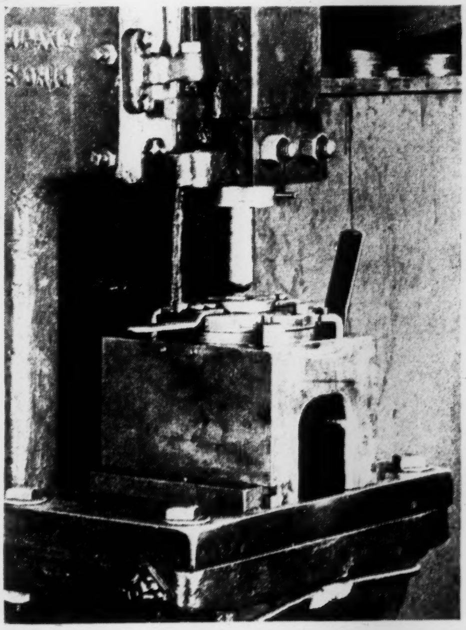

FIG. 2.—Cup-Drawing Equipment Assembled in the Punch Press.

Figure 1 is a sketch of the equipment used in the cup-drawingtest, and Fig. 2, shows the equipment assembled in the punch press. This tool differs from the types used by other investigators (**8, 9, 10, 11**) chiefly in the provisions made to eliminate the effect of hold-down pressure on the test results. As shown in Fig. 1, tapered relief is provided in the cap of the die to allow for the progressive thickening of the flange of the specimens during drawing. The gradual increase in clearance toward the center of the die is based on the theoretical amount of thickening accompanying the reduction in diameter, due to upsetting.

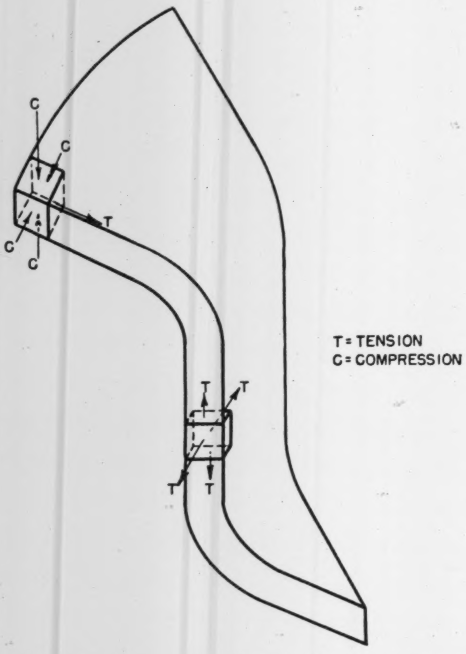

FIG. 3.—Stress in Two Locations in Wall of Partially Formed Cup-Drawing Test Blank.

In making a test, the blanks are placed on the die and centered by means of spacer rings. The cap of the die is placed in contact with the blank at its circumference and held in this position by means of set screws adjusted by finger pressure. The punch is then forced through the die producing a deep-drawn cup or fracturing the blank. If the drawing operation is successful, the return movement of the punch strips the cup against the bottom rim of the die. Different dies are employed for testing different gages of material so that the annular clearances are comparable. The three dies used for the tests were designed to give about 21 per cent annular clearance on stock 0.050, 0.043, and 0.037 in. in thickness. The respective diameters of the dies are 1.121, 1.105 and 1.090 in., and the drawing radius is $\frac{5}{16}$ in. A plunger 1.000 in. in diameter is used with all dies. Lard oil, applied to the test blanks by swabbing, was used as a lubricant to reduce friction and scoring during the drawing operation.

Figure 3 shows the stresses occurring in the specimens during the testing operation. The principal stresses during the first movement of the punch are those of bending around the nose of the punch and the radius of the die, with a certain amount of circumferential compression also acting. As the blank moves around the radius and side of the punch, there is a movement of metal inward toward the center produced by tensile stresses. The metal in the flange is subject chiefly to radial tension and circumferential compression. A slight amount of vertical compressive stress is also acting but this is limited because of the radial taper in the cap. This brief outline of the operation indicates that the cup-drawing test simulates at least some of the actions involved in all deep-drawing operations.

Preliminary tests with the cup-drawing equipment were made with an Amsler tension testing machine used as a press. Since drawing loads could be measured with this equipment, the tests provided considerable information about the testing method. This work showed that breakage figures were readily reproducible within 10 per cent. The tests showed that blanks that were about 0.03 larger than the critical diameter invariably showed 100 per cent breakage in the tests. Blanks about 0.03 in. smaller than the critical diameter were formed into cups with zero breakage.

The tests with the Amsler equipment also showed that the load required to produce drawing, increases with blank diameter as long as the cups can be formed. Failure of blanks larger than the critical size, however, always occurs at loads lower than those withstood by smaller disks. Fractures of the specimens ordinarily occur at the junction of the dome and the cylindrical sides of the cups. These facts indicate that the failures are caused by an inability to contract the circumference of the blanks at loads low enough to escape serious necking in this region. Thus, the fractures of the specimens are not caused primarily by simple stretching but result from improper distribution of the stresses.

The tests with the Amsler machine also indicated that increased drawing speeds between 1 and 20 in. per min. raise the loads required for cup-drawing. The effect of drawing speeds in this particular range on breakage was not significant. Later, it was determined that the breakage figures for drawing at 3 in. per min. on the Amsler machine and at 100 in. per min. on a punch press were essentially identical for tests on four representative steels.

Since the work indicated that the drawing loads had no value in rating drawability, a punch press was substituted for the testing machine used in the early work. The tests discussed in this paper were made on a standard crank-operated press. The machine has a plunger speed of about 100 in. per min. and a stroke of 3 in. The blanks tested were also made on the punch press by the use of interchangeable blanking dies. The burring on the edges of blanks was very slight. Previous tests made on machined blanks gave results identical with those obtained on sheared blanks, indicating that shearing was a satisfactory method of preparing specimens. The surface condition is believed to have been similar for all steels because they were all finished on the same temper rolls.

The results obtained in this test are reported in terms of "cup-drawing indices." The cup-drawing index is made up of two parts. The first part of the value is a figure denoting the size of the critical diameter as the number of $\frac{1}{32}$-in. increments over 2 in. The last part of the index indicates the percentage success in forming blanks of the critical size for the material. For example, a steel may be said to have a cupping index of 9–60 per cent. Its drawing quality as measured by this test may also be described at length as follows: 40 per cent of the 2 in. plus $\frac{9}{32}$-in. blanks broke and 60 per cent formed cups successfully, all blanks of smaller diameter formed cups successfully and all blanks over $2\frac{9}{32}$ in. in diameter broke on testing.

TABLE I.—CUP-DRAWING INDICATOR NUMERALS FOR BLANKS OF VARIOUS SIZES USED IN TESTING SHEET STEELS.

Cup-Drawing Number or Index	Blank Diameter, in.		Reduction, per cent or $\frac{\text{Blank Diameter} - \text{Cup Diameter}}{\text{Blank Diameter}}$
	Fractions	Decimals	
0 (base size)	2+ 0	2.000	50.0
1	2+ 1/32	2.031	50.8
2	2+ 2/32	2.062	51.5
3	2+ 3/32	2.094	52.3
4	2+ 4/32	2.125	53.0
5	2+ 5/32	2.156	53.6
6	2+ 6/32	2.187	54.3
7	2+ 7/32	2.219	55.0
8	2+ 8/32	2.250	55.5
9	2+ 9/32	2.281	56.1
10	2+10/32	2.312	56.8
11	2+11/32	2.344	57.3
12	2+12/32	2.375	57.8
13	2+13/32	2.406	58.4
14	2+14/32	2.437	59.0
15	2+15/32	2.469	59.5

Table I gives a list of the numerals assigned to various blank sizes. The scale is based on 2-in. diameters because this is the minimum ordinarily encountered in drawing quality steels. Intervals of $\frac{1}{32}$ in. are employed because they are satisfactory for measuring small variations in drawing performance and make the scale almost continuous. The reproducibility of the breakage figures is of a high order and makes it possible to use them in making fine differentiations in drawing quality.

TABLE II.—AGREEMENT OF ACTUAL DRAWING PERFORMANCE AND CUP-DRAWING TEST RESULTS.

Part	Steel	Breakage, per cent	Olsen	Elongation in 2 in., per cent	Cup-Drawing Index, per cent
Unknown	A	1.2	0.383	30	6-100 (7-0)
	B	8.4	0.360	33	6-60
Unknown	III	1.8	0.443	44.5	10-80
	I	5.0	0.444	39.5	10-60
Front fender	B4	2	0.413	44	8-30
	B3	6	0.423	32.5	6-50
Rear fender	1A	3.3	0.460	43.5	10-90
	2A	5.4	0.409	37	5-100 (6-0)
Quarter panel	3A	[a]	0.449	41.5	8-55
	4A	[a]	0.456	41.5	9-100 (10-0)
	5A	[a]	0.442	43.5	7-60

[a] No breakage figures reported but press-shop foreman stated that he considered steel 4A to be the best of the three in this group.

COMPARISON OF CUP-DRAWING RESULTS WITH PERFORMANCE DATA

A few data showing that the results of cup-drawing tests are in agreement with the performance of materials in actual drawing operations were obtained early in the investigation. Some of these results are given in Table II and represent plant tests on different steels in the same operations. In every case, the steel which had the higher cup-drawing value had less breakage in the same press operation. The first two groups apparently illustrate conditions where slight variations in sheet quality are important. Although the forming conditions are unknown, it seems likely that they were very severe for the grades of steel involved. Hence, the slight superiorities in quality, indicated by the small differences in cupping values, were sufficient to avoid excessive breakage. It is unfortunate that more samples with known service histories could not be tested, but they were difficult to obtain because of the cessation of production of severely deep-drawn automotive parts. Because of the lack of opportunity for correlation with service, the method cannot be evaluated for its reliability and usefulness, or the reverse, in appraising what the performance of a given lot will be. Hence, it is presented as one more means of studying steels for deep-drawing operations which might ultimately evolve into one of several acceptance tests. The different kinds of tests needed, and the weight to be given to each kind will vary with the type of deep-drawing the steel must withstand.

TABLE III.—SHOWING LACK OF RELATIONSHIP BETWEEN CUP-DRAWING RESULTS AND STANDARD TESTS.

Steel	Cup-Drawing Index, per cent	Rockwell B Hardness	Olsen Ductility Value, in.	Elongation in 2 in., per cent
No. 209	11-65	41	0.413	37.5
No. 114	11-20	32	0.429	40.5
No. 208	10-65	42	0.413	38.5
No. 213	10-20	44	0.407	36.5
No. 88	9-100 (10-0)	39	0.407	41.0
No. 92	9-100 (10-0)	39	0.412	42.5
No. 98	9-100 (10-0)	41	0.427	41.5
No. 99	9-45	36	0.409	41.5
No. 107	8-90	42	0.444	43.0
No. 105	8-50	42	0.451	43.0
No. 161	7-100 (8-0)	42	0.413	47.0
No. 62	7-100 (8-0)	43	0.445	38.5

In the data given in Table II it is noticeable that the cup-drawing values do not always follow the results of the Olsen and elongation tests. Additional data showing that the cup-drawing test measures a property that is not evaluated by the hardness, Olsen, and tension tests are given in Table III. Steel No. 209, for example, had the highest cup-drawing value, although it had low Olsen and percentage elongation values. Conversely steels Nos. 105 and 107 had high Olsen figures and steels Nos. 161 and 107 had high elongation values combined with average cup-drawing indices. Similarly the other data show wide discrepancies in the ratings made by the four testing methods on all of the samples.

TESTS ON COMMERCIAL-RIMMED STEELS

Over 200 samples of commercial steels made for deep-drawing applications were given the ordinary standard tests and tested in the experimental cup-drawing equipment. The samples represented 142 different heats, and chemical analyses were obtained on all materials.

The basic data are not included, but in order to present the data in a concise manner, the results have been considered on a statistical basis. The distribution of the ratings given the samples by the cup-drawing, Olsen, and Rockwell "B" hardness tests are shown in Fig. 4 by means of frequency polygons.

The close resemblance in the shape of the polygons shows that the frequency distributions in the three tests are similar. This is the expected distribution for any group of ostensibly similar samples when the test methods are reliable and the quality of the material is affected by a large number of variables. For all three of the test methods, the modal or most frequently occurring value is near the average of the whole group. The similarity of the frequency distributions indicates that the cup-drawing test is as consistent in measuring a property of the group as the standard Olsen and Rockwell hardness tests.

As illustrated in Fig. 4, the average cup-drawing index of all the samples of rimmed steels tested is 7–85 per cent, and the most frequently occurring index is given as 7–75 per cent. Over two thirds of the samples tested had indices above 7–50 per cent.

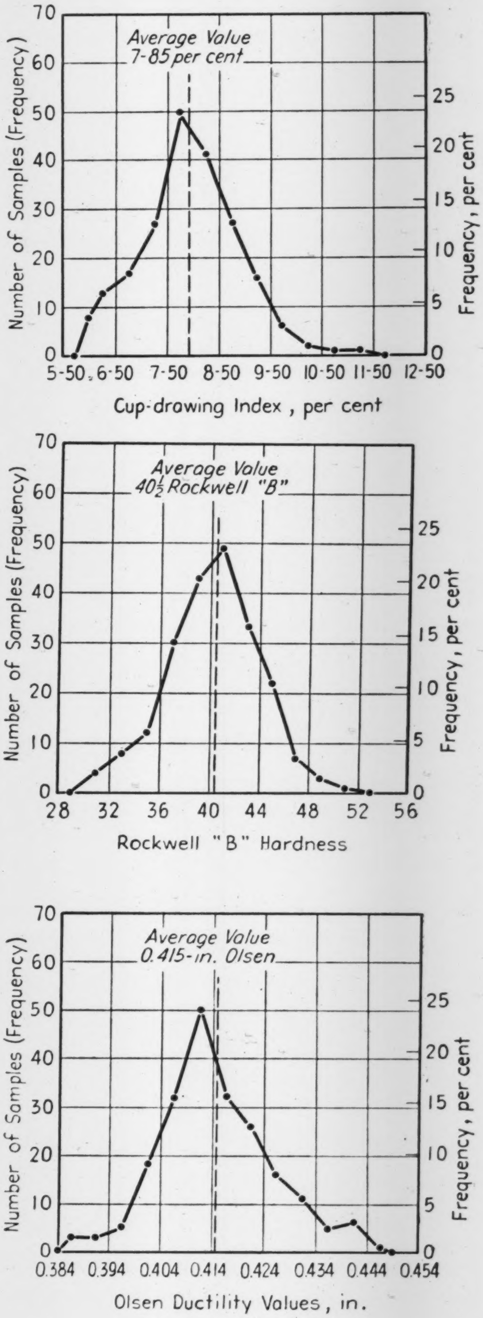

FIG. 4.—Frequency Polygons for Cup-Drawing, Hardness, and Olsen Values on 212 Rimmed-Steel Samples.

The frequency polygon for the Rockwell B hardness values shows that the average is 40½. The most frequently obtained hardness reading was 41 Rockwell B hardness. About 71 per cent of the samples had hardness values ranging from 36 to 44.

The bottom polygon in Fig. 4 shows the frequency distribution for the Olsen ductility values. The average value is 0.414 and the most frequent reading obtained was 0.413. Approximately two thirds of the samples had ductility ratings between 0.404 and 0.424. The regular distribution of the Olsen readings is partly caused by the fact that the sample group contained appropriate proportions of different gages. As many investigators have pointed out, the effect of gage is important in the Olsen test, and presumably the distribution would have been slightly different if all the samples had been the same thickness.

TABLE IV.—AVERAGE CUP-DRAWING INDICES FOR GROUPS OF TEN SAMPLES ARRANGED IN SUCCESSIVE ORDER OF OTHER TEST VALUES.

Group	Rockwell B Hardness		Olsen		Elongation in 2 in.		Yield-Point Elongation	
	Average Hardness	Average Cupping Index, per cent	Average Olsen	Average Cupping Index, per cent	Average Elongation, per cent	Average Cupping Index, per cent	Average Elongation, per cent	Average Cupping Index, per cent
No. 1	32.7	7-83	0.394	7-28	29.8	7-45	0.35	7-55
No. 2	35.1	8-54	0.402	7-77	35.5	7-34	1.25	7-75
No. 3	36.5	8-08	0.405	8-52	37.4	8-00	1.40	8-20
No. 4	37.6	8-14	0.406	7-35	38.0	7-66	1.75	8-29
No. 5	38.0	8-19	0.407	8-13	38.9	8-13	1.75	7-94
No. 6	38.7	7-98	0.409	7-87	39.0	7-40	2.00	7-83
No. 7	39.0	8-40	0.410	7-58	39.5	7-37	2.25	7-82
No. 8	39.1	7-78	0.411	7-30	40.0	7-59	2.25	7-38
No. 9	40.0	7-74	0.412	7-96	40.0	8-00	2.25	7-31
No. 10	40.6	8-09	0.413	8-10	40.0	7-57	2.75	8-52
No. 11	41.0	8-44	0.414	7-58	40.9	8-04	2.75	7-95
No. 12	41.9	7-75	0.415	7-75	41.0	8-46	2.75	7-97
No. 13	42.0	8-22	0.417	8-25	41.0	8-03	2.85	7-70
No. 14	42.0	7-60	0.419	7-55	41.6	8-71	3.25	9-00
No. 15	42.9	8-00	0.421	8-02	42.0	8-92	3.25	7-92
No. 16	43.0	8-07	0.422	7-82	42.0	7-66	3.25	7-68
No. 17	43.5	7-76	0.425	8-25	42.1	7-76	3.75	8-30
No. 18	44.6	7-21	0.428	8-84	43.0	8-22	3.75	7-76
No. 19	45.5	7-28	0.431	8-75	43.3	8-01	4.05	7-69
No. 20	47.1	7-76	0.439	7-82	45.1	8-15	4.70	8-00
Number of Samples	203		203		201		201	

Similar frequency distributions were found for the yield point, tensile strength, and elongation values from the tension tests.

Lack of Correlation of Test Results:

In spite of the fact that the cup-drawing, Olsen, and hardness tests agree in the general distribution of test values for the group, there is no close agreement on the ratings given individual materials. Since it would be confusing to present the data in the form of scatter charts based on individual readings, the results were grouped according to the values obtained in the various tests. The average cup-drawing indices for groups of ten samples arranged in the order of other test results are given in Table IV. Because mechanical tests are supposed to

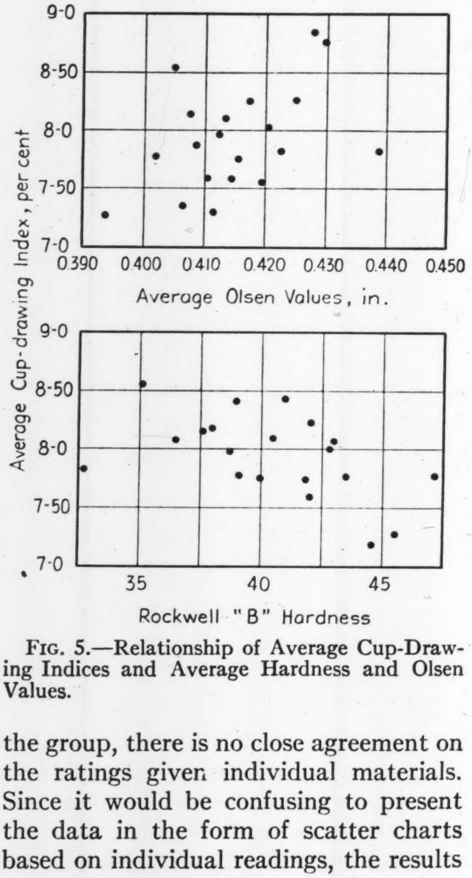

FIG. 5.—Relationship of Average Cup-Drawing Indices and Average Hardness and Olsen Values.

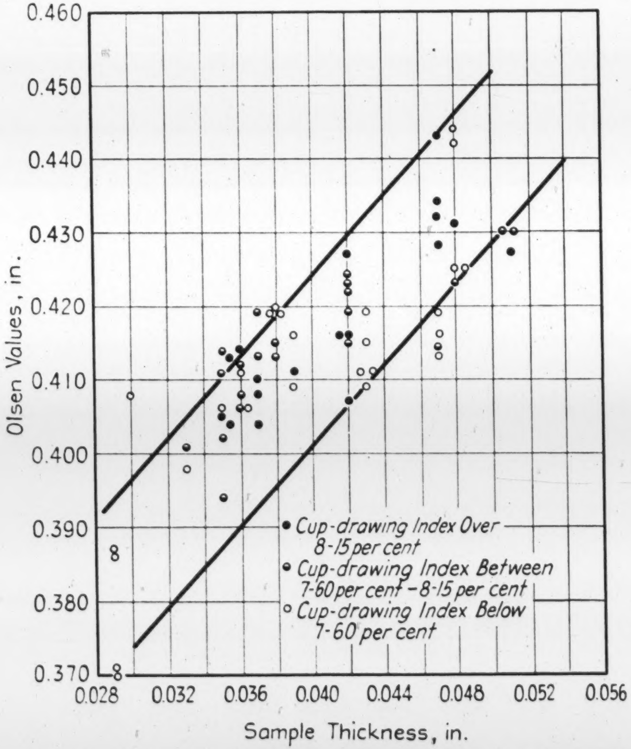

FIG. 6.—Illustrating the Effect of Gage on Olsen Values and the Independence of Cup-Drawing Indices of Thickness and Olsen Ratings.

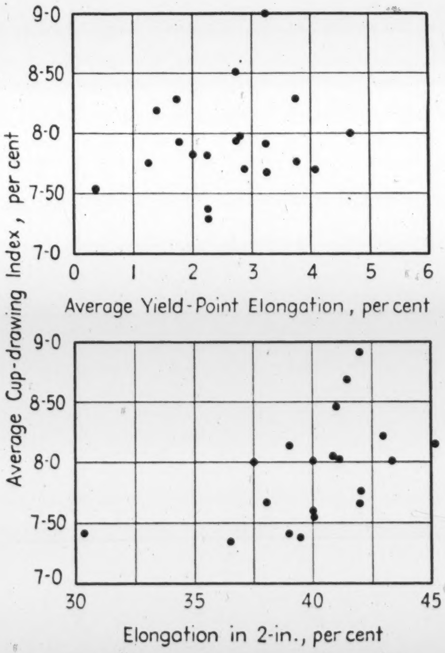

FIG. 7.—Relationship of Average Cup-Drawing Indices and Average Values Obtained in the Tension Tests.

measure the integrated result of differences in production variables, the data were not segregated on the basis of the known variables.

The average cupping indices for the groups of ten samples are plotted against different ranges of Rockwell hardness and Olsen values in Fig. 5. The distribution of the points shows a slight tendency for the association of lower cup-drawing indices with hardness values over 44 Rockwell B hardness. The scatter is so great, however, that the agreement must be considered to be of only a general nature.

TABLE V.—LIST OF BEST AND POOREST SAMPLES TESTED.
Classification Based on Cup-Drawing Indices.

Sample	Heat	Lift	Carbon, per cent	Sulfur, per cent	Temper Rolling, per cent	Rockwell B Hardness	Olsen Value	Yield-Point Elongation, per cent	Elongation in 2 in., per cent	Yield Strength, psi.	Tensile Strength, psi.	Cup-Drawing Index, per cent
						BEST SAMPLES						
No. 114	No. 31 550	No. 393	0.07	0.028	0.37	32	0.429	1	40.5	22 750	45 750	11–20
No. 41	No. 32 638	No. 1564	0.08	0.025	0.40	33	0.424	3.25	42.0	29 250	43 500	10–40
No. 40	No. 32 638	No. 1574	0.08	0.025	0.40	36	0.429	3.25	42.0	31 250	43 750	10–30
No. 46	No. 36 542	No. 272	0.08	0.026	0.40	41	0.421	2.75	39.0	29 750	45 250	10–0 (9–100)
No. 47	No. 36 542	No. 271	0.08	0.026	0.40	36	0.430	2.25	39.5	28 500	44 500	10–0 (9–100)
No. 88	No. 30 377	No. 9223	0.07	0.027	0.17	39	0.407	3.50	41.0	32 250	44 750	10–0 (9–100)
No. 92	No. 19 990	No. 4432	0.06	0.024	0.44	39	0.412	1.75	42.5	30 750	46 750	10–0 (9–100)
No. 98	No. 31 525	No. 9253	0.08	0.025	0.37	41	0.427	1.75	41.5	30 000	47 000	10–0 (9–100)
No. 45	No. 36 542	No. 263	0.08	0.026	0.40	37	0.430	3.25	40.5	31 250	45 750	9–90
No. 99	No. 27 948	No. 9321	0.08	0.027	0.17	36	0.409	4.75	41.5	35 250	45 000	9–45
Average			0.076	0.026	0.35	37	0.422	2.75	40.7	30 100	45 200	10–15
						POOREST SAMPLES						
No. 32	No. 25 444	No. 2601	0.08	0.024	0.90	40	0.415	1.50	40.0	35 000	46 750	6–0 (5–100)
No. 144	No. 28 734	No. 6223	0.08	0.026	0.71	31	0.411	2.25	42.5	39 250	45 000	6–0 (5–100)
No. 145	No. 28 734	No. 6224	0.08	0.026	0.71	32	0.406	2.25	42.5	31 250	46 000	6–0 (5–100)
No. 152	No. 28 734	No. 6225	0.08	0.026	0.23	34	0.412	2	39.5	30 750	46 250	6–0 (5–100)
No. 153	No. 18 365	No. 6049	0.08	0.026	1.18	44	0.413	2.25	39.5	35 750	49 250	6–0 (5–100)
No. 217	No. 18 406	No. 3972	0.09	0.028	0.91	33	0.409	0	39.5	28 750	45 000	6–0 (5–100)
No. 218	No. 18 406	No. 3970	0.09	0.028	0.91	37	0.411	1	40.5	32 750	46 000	6–0 (5–100)
No. 151	No. 28 734	No. 6224	0.08	0.026	0.23	36	0.410	2.25	39.5	32 500	47 500	6–10
No. 227	No. 21 830	No. 3311	0.08	0.028	1.13	39	0.409	3.50	39.0	36 500	46 750	6–10
No. 31	No. 17 998	No. 2608	0.08	0.024	0.90	40	0.419	1.25	40.5	33 500	45 500	6–20
Average			0.082	0.0264	0.78	36.5	0.411	1.9	40.4	32 600	46 300	6–05

The distribution of the averages for the Olsen test and the cup-drawing values is so erratic that it is evident that there is no agreement between the results of the two methods. One reason for the lack of correlation between the Olsen and cup-drawing test results on the same materials is illustrated in Fig. 6. This figure shows the dependence of Olsen values upon the thickness of the sample tested. It also shows the tendency for heavier gage samples to give higher Olsen readings. Different symbols are used to identify individual samples which had poor, average, and superior cup-drawing indices. The locations of the points show that high cup-drawing values are found in all thicknesses and at all Olsen levels. The independence of the cup-drawing test of thickness of samples tested is accomplished, of course, by the use of different dies for materials of different gages. This procedure is in line with commercial drawing operations.

A plot of the average cup-drawing values against the elongation figures from tension tests is shown in Fig. 7. The top part of the figure shows that there is no relationship between the cup-drawing behavior of the samples and the severity of aging as reflected by the yield-point elongation in the tension test. Since yield point elongation is a criterion of aging this behavior suggests that the superior drawing properties of killed steels may not be directly associated with the fact that they are less prone to age than rimmed steels.

The lower chart in Fig. 7 shows that the groups of samples with greater tensile elongation values gave better cup-drawing performances. There is considerable scatter in the results because of the differences inherent in the testing methods. The relationship is general rather than precise, and very often samples which have high elongation figures possess low cupping indices.

When the samples which had unusually good or unusually poor cup-drawing indices are grouped together, it is even more apparent that the various tests rate the samples on different bases. Table V lists the ten best and ten poorest samples in the groups according to their cupping performances. The values obtained in the tension test and the Olsen and hardness figures are also given for the individual samples.

The disparity in cup-drawing values for the two groups is pronounced, although the hardness and tension elongation figures for the two groups are closely similar. The sheets with the best cup-drawing properties usually had higher Olsen values than the samples in the poorer group, although this was not true in every case. The difference in average Olsen values (0.422 to 0.411), however, fails to suggest the wide variation in quality shown by the cup-drawing test.

Table V indicates that when individual samples are being considered the cup-drawing test integrates properties not reflected by the other tests.

EFFECT OF COMPOSITION ON CUP-DRAWING INDICES

The data in Table V show that five of the ten samples which gave the best performance in the cup-drawing test were from two heats—Nos. 32,638 and 36,542. Similarly, six of the ten poorest sheets came from only two heats—Nos. 28,734 and 18,406. Since these heats were not outstanding on the basis of composition, they seem to illustrate the importance of the individual heat and processing histories in connection with cup-drawing results. The data in Table V also show that the group with the poorest cup-drawing indices had slightly higher average carbon and sulfur contents than the other group.

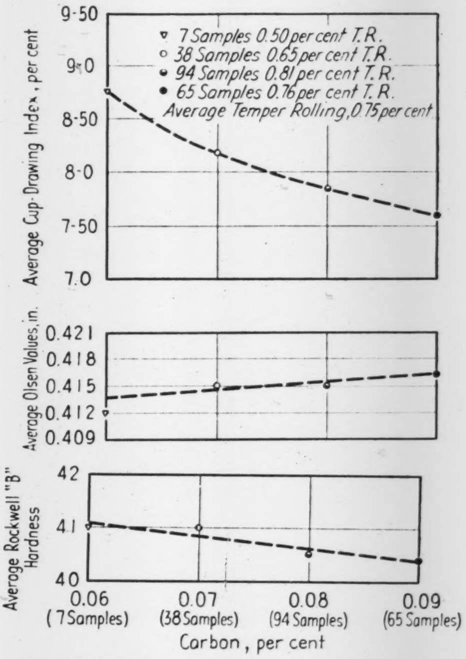

FIG. 8.—Effect of Carbon Content on Cup-Drawing Olsen and Hardness Test Results.

In order to determine more extensively the effect of different elements on cup-drawing performance, the samples were divided into subgroups on the basis of chemical composition. Since a fairly large number of samples make up each group, the differences between the subgroups are largely attributable to the main variable under consideration.

In the initial examination of the data, the test results were grouped on the basis of the carbon content of the samples. Figure 8 shows the effect of average carbon content on the average Olsen, hardness, and cup-drawing test results. Symbols in this figure indicate the average amount of reduction in temper rolling for the different groups.

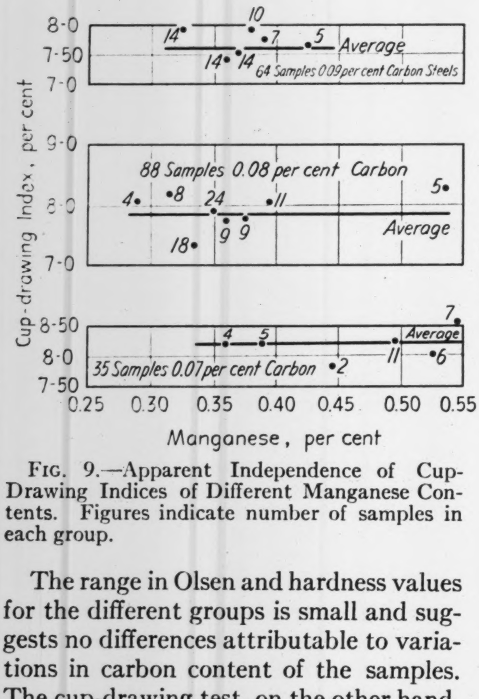

FIG. 9.—Apparent Independence of Cup-Drawing Indices of Different Manganese Contents. Figures indicate number of samples in each group.

The range in Olsen and hardness values for the different groups is small and suggests no differences attributable to variations in carbon content of the samples. The cup-drawing test, on the other hand, shows a marked difference in the average behavior of steels of different carbon contents. For the samples tested, any increase in carbon content significantly and uniformly lowered the average cup-drawing value. The average cup-drawing indices range from 8–75 per cent for the 0.06 per cent carbon group to 7–60 per cent for the 0.09 per cent carbon group. Because a large number of samples were tested, and the groups are known to be similar except for carbon content, the results appear to be an accurate reflection of the effect of this element. The effect of carbon in reducing the cup-drawing rating agrees with opinions, based on practical experience, of its probable effect on drawability.

In order to eliminate the effect of carbon while studying the effect of manganese, the samples were first divided into three carbon groups. Figure 9 shows the average cup-drawing indices for different manganese ranges of the 0.07, 0.08, and 0.09 per cent carbon steels. The number of samples in each subgroup is marked near each point on the graph. Since the results indicate that variations in manganese content, within the range studied, have little effect on average cupping values, only average values are marked on the chart.

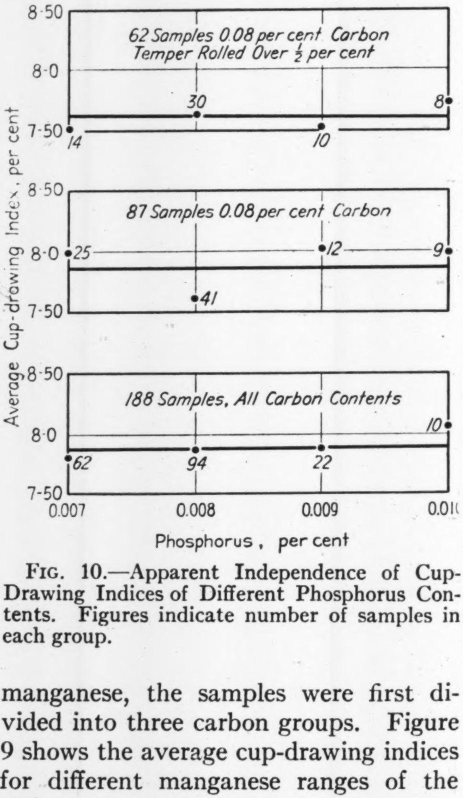

FIG. 10.—Apparent Independence of Cup-Drawing Indices of Different Phosphorus Contents. Figures indicate number of samples in each group.

Figure 10 shows that the phosphorus content of the samples tested had little effect on the average cup-drawing indices. This is illustrated by the plots for all the samples, for the samples of the same carbon content, and also for the samples subdivided on the additional basis of severity of temper rolling. It might be expected that poorer drawing quality would be associated with higher phosphorus contents, but the data show no effect on cup-drawing indices. The variation in phosphorus content ranged only from 0.006 to 0.010 per cent and such small amounts probably have correspondingly small effects on mechanical properties.

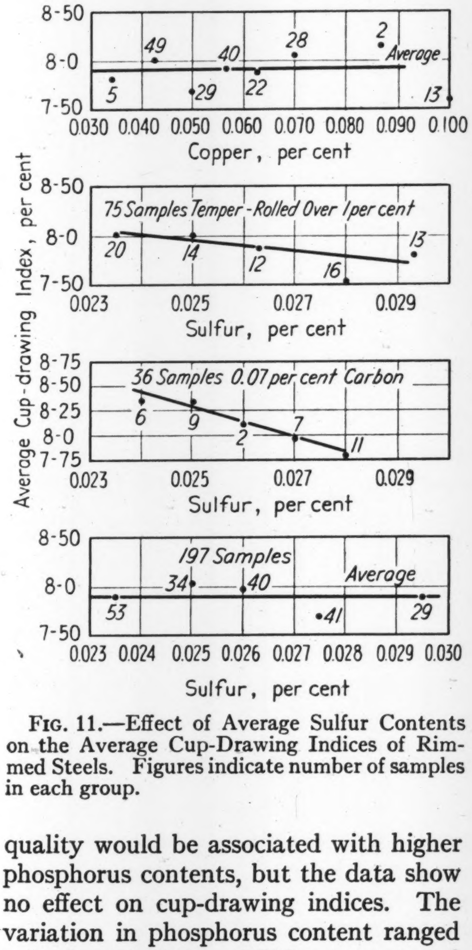

FIG. 11.—Effect of Average Sulfur Contents on the Average Cup-Drawing Indices of Rimmed Steels. Figures indicate number of samples in each group.

Figure 11 shows the average cup-drawing indices for the rimmed steels when the results are grouped according to the sulfur and copper contents of the samples.

In the top section of Fig. 11, showing the effect of copper, the average values are irregularly distributed around the mean cupping index. The location of the points indicates that there is no direct relationship between the copper contents of the samples and their cup-drawing behavior. This agrees with published data on the effect of copper, in this range, on the deep-drawing performance of sheet steels.

The other plots in Fig. 11 show the effect of sulfur on the average index of rimmed steels. When all the samples are considered, the points are distributed fairly close to the average value. Since there is abundant evidence to indicate that high sulfur contents are deleterious in deep-drawing steels, the data were subdivided according to other variables. The other two plots show that increased sulfur contents do lower the cup-drawing ratings when the effects of carbon content and temper rolling are eliminated by segregating the data.

These plots show that increasing sulfur contents from 0.023 to 0.030 per cent lower the average cup-drawing index for the 0.07 per cent carbon samples from 8-35 per cent to 7-80 per cent. Similarly, for samples which had been temper rolled over 1 per cent, a decrease in cupping index from 8-0 to 7-80 per cent is found in the higher sulfur content. The magnitude of the change in cup-drawing performance is believed to be of the order of the decreased press performance for sulfur variations of this range.

EFFECT OF TEMPER ROLLING ON CUP-DRAWING INDICES

In addition to studying the data in the light of composition variables, an attempt was made to determine the effect of differences in the severity of temper rolling. For this comparison the average values for the hardness, Olsen, and cup-drawing tests were grouped according to three ranges of temper rolling. The ranges used are indicated in the graphs used to illustrate the relationships found. Since carbon content had been shown to be a significant variable affecting cup-drawing performance, a further subdivision was made on the basis of carbon contents.

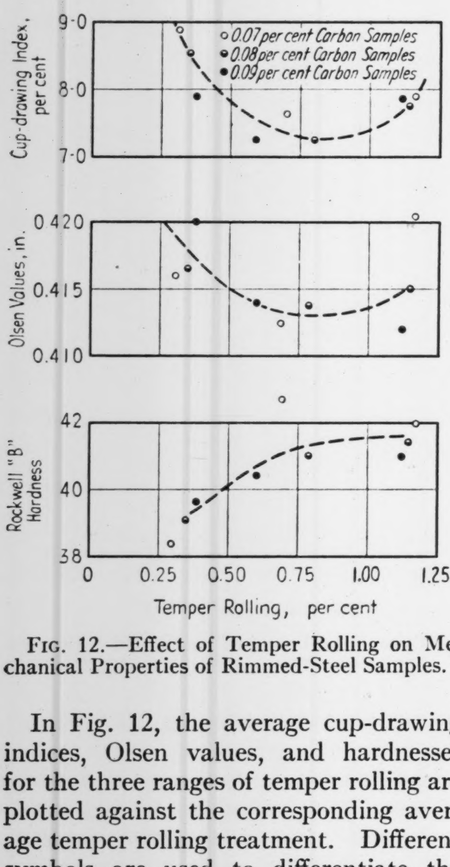

FIG. 12.—Effect of Temper Rolling on Mechanical Properties of Rimmed-Steel Samples.

In Fig. 12, the average cup-drawing indices, Olsen values, and hardnesses for the three ranges of temper rolling are plotted against the corresponding average temper rolling treatment. Different symbols are used to differentiate the carbon groups, but since the general behavior is similar, the curves correspond to the weighted averages.

The bottom curve indicates that the variations in temper rolling had little effect on the Rockwell "B" hardness of the samples. The hardness averages increase slightly with increasing severity of temper rolling. The rate of increase in hardness is greater for amounts between 0.30 and 0.70 per cent than it is between 0.70 and 1.15 per cent.

The location of the points for the different carbon groups shows that the average hardness of the 0.07 per cent carbon group is greater than the others for the samples rolled over 0.50 per cent. For samples rolled less than this amount, the hardness is in the order of the carbon contents. The top curves show that this variation in the effect of temper rolling is not mirrored by the cup-drawing indices or the Olsen values. The Olsen values, however, are in the same order as the hardness averages on the basis of the carbon content except for the samples rolled between 0.5 and 1 per cent.

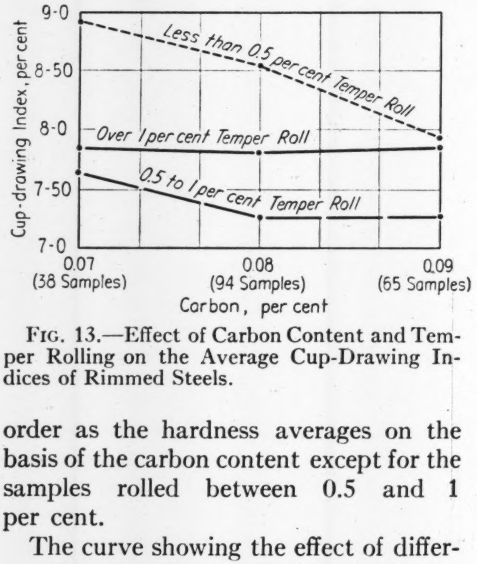

FIG. 13.—Effect of Carbon Content and Temper Rolling on the Average Cup-Drawing Indices of Rimmed Steels.

The curve showing the effect of different degrees of temper rolling on the average cup-drawing index is shown at the top of Fig. 12. All of the carbon groups show a minimum cup-drawing index in the intermediate range of temper rolling. The indication of lower ductility for these groups is also shown by the Olsen results on the 0.07 and 0.08 per cent carbon steels. The cup-drawing results seem to indicate that samples of rimmed steels temper rolled in the range of 0.5 and 1.0 per cent have poorer drawing qualities than those rolled in smaller amounts. In this respect, it is worthy of mention that samples of good drawing performance listed in Table V were all temper rolled less than 0.44 per cent. The ten samples of the group showing poor drawing qualities had been temper rolled an average of 0.78 per cent.

The magnitude of the changes in the average cup-drawing values for the different carbon groups is shown more clearly in Fig. 13. The graph indicates that the carbon content is the controlling variable in samples rolled less than 0.5 per cent, and that the lower carbon group has the better average cupping index. For the samples rolled over 1 per cent, the cup-drawing indices were practically unaffected by the carbon contents of the groups. For the groups which received the intermediate amounts of temper rolling, the 0.08 and 0.09 per cent carbon samples had identical average cup-drawing indices. This average index was considerably lower than the average for the 0.07 per cent carbon samples rolled similar amounts. These results indicate that temper rolling impairs the drawing qualities of rimmed steels. In general, the deleterious effects of temper rolling are less noticeable with increasing carbon contents, and the 0.08 and 0.09 per cent carbon steels are similar in drawability when temper rolled 0.5 per cent or more. It also appears that for the samples tested, temper rolling in amounts of about 1 per cent or more masks differences initially attributable to variations in carbon content.

CONCLUSIONS

The results of tests on approximately 200 samples of commercial rimmed sheet steels indicate:

1. The results of the cup-drawing test have a high order of reproducibility.
2. The experimental test method rates materials in a different manner than the standard ductility, hardness, and tension tests.
3. The properties measured by the cup-drawing test are believed to be some of those which control the deep-drawing performance or drawability of materials. The few data available from commercial operations support this opinion.
4. The average effect of variations in composition on cup-drawing test results, and presumably drawability, were determined for the samples tested. Increases in carbon and sulfur contents were found to lower cup-drawing indices. Variations in manganese, copper, and phosphorus contents, for the ranges covered by the sample group, were found to be without effect on the test results.
5. The data indicate that temper rolling lowers the cup-drawing index and that the effect is most pronounced in the neighborhood of 0.75 per cent reduction or in the range of 0.5 to 1 per cent.

Acknowledgments:

The work reported in this paper was performed in the laboratories of the Battelle Memorial Institute under the sponsorship of the Inland Steel Co. The authors wish to thank C. E. Sims, and O. E. Harder of Battelle Memorial Institute, C. A. Mahlie and J. H. Nead, of Inland Steel Co., and George Sachs of the Case School of Applied Science for their counsel and cooperation.

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DISCUSSION

Mr. J. R. Low[1] (*presented in written form*).—On the basis of the results given in Table II of the paper by Messrs. Boulger and Dahle, it appears that the test described may have considerable merit, since in every case the test results agree qualitatively with the press performance. It is unfortunate that more data of this type are not available, however, since these same results suggest that for some drawing operations, at least, the test lacks sensitivity. For example, steels A and B with breakages of 1.2 per cent and 8.4 per cent have drawability indices of 6–100 (7–0) and 6–60, respectively; on the other hand, steels 1A and 2A with breakages of 3.3 per cent and 5.4 per cent have drawability indices of 10–90 and 5–100 (6–0). In the first case, the considerable difference in press performance is not indicated by the test results, whereas in the second case a relatively smaller difference in press performance corresponds to widely different drawability indices.

On page 512 the authors state that there is no apparent relation between susceptibility to aging and drawability as measured by this test, basing their statement on the fact that there is no systematic relation between yield-point elongation and drawability index. While it is true that the increase in yield-point elongation with time of temper-rolled steels may be used as an indication of susceptibility to aging it would seem more reasonable to compare the steels on the basis of the stress at the yield point since this value is more directly related to the tensile stresses in the cup wall which lead to failure. This objection is supported to some extent by the results shown in Table V in which the majority of the yield-strength values for the "best" steels are consistently lower than those for the "poorest" steels. Further, the elapsed time between temper rolling and tension testing as well as the temperature prevailing during this time may affect the yield-point elongation, and if these two variables were not substantially constant it does not follow that the yield-point elongation may be taken as a measure of aging susceptibility.

In view of the fact that other investigators[2,3,4] have used a very similar testing method without obtaining as high a degree of correlation between press performance and test results, it would seem that additional verification of the authors' results is needed. The authors' modification of the usual hold-down fixture and the use of a punch of large radius may very well have eliminated the principal causes of inconsistent results in this type of test, and it is to be hoped that more steels of known press performance will be made available for testing.

Mr. F. W. Boulger[5] (*by letter*).—The authors appreciate Mr. Low's discussion which indicates the care with which he read the paper and agree that additional tests on materials of known service history would be desirable. An effort will certainly be made to secure such information when the opportunities arise.

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[1] Assistant Professor, Department of Metallurgy, Pennsylvania State College, State College, Pa.

[2] G. Oehler, "The Elimination of Scrap in Drawing Hollow Bodies of Thin Sheets with Special Reference to the Available Deep Drawing Tests," *Beiträge zur Wirtschaft, Wissenschaft und Technik der Metallen und Ihre Legierungen*, No. 5, pp. 1-81, N. E. M. Verlag, Berlin (1938).

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A. G. C. Gwyer and P. C. Varley, "A Deep Drawing Test for Aluminum," *Journal*, Inst. Metals, Vol. 58, p. 83 (1936).

[4] A. Pomp and A. Krisch, "Deep Drawing Experiments with Sheet and Strip of Alloy Steels," *Mitteilungen aus lem, Kaiser-Wilhelm Institut für Eisenforschung zu Dusseldorf*, Vol. 22, pp. 19–34 (1940).

[5] Research Metallurgist, Battelle Memorial Institute, Columbus, Ohio.

The variations between breakage figures in service and in the test are difficult to explain because of ignorance of all the conditions involved. It is possible that the differences might be explained by sampling variations and that the sheet tested was better or worse than the lift from which it came. On the other hand, it seems likely that slight differences in quality are more critical in some drawing operations than in others.

In regard to the observations on aging criteria, we should like to point out that yield-point elongation was used for an index of severity of aging because it was the only one available. Since the yield strength in the freshly rolled condition was unknown the increases caused by aging could not be determined on that basis.

As pointed out by Mr. Low, the yield strengths of the ten best steels were usually lower than the yield strengths of the ten worst steels. However, the original data not given in the paper show that 140 of the 210 steels tested had yield strengths higher than the average of the ten worst steels. Of these, 92 had cup-drawing ratings superior to 7–50 per cent, the most frequently occurring value for the group. These facts, together with scatter charts plotted when the tests were made, show that no close agreement was found between yield strength and experimental test values. Yield strengths would not be expected to affect drawing performances directly because breakage only occurs after the ultimate strength has been exceeded.

MR. J. J. SHUMAN.[6]—I suggest that the author state his definition of yield-point elongation as used in the paper. The term seems new to me and probably is to others, and I believe it is not included in the Society's standard definitions.

MR. M. F. RUPP.[7]—I would like to ask whether the authors have done any work on other metals than steel, for example, on non-ferrous metals. The aircraft people are interested in drawability, formability, and workability. I think it would be an excellent test.

MESSRS. FRANCIS W. BOULGER and F. B. DAHLE[8] (*authors' closure, by letter*).—In response to Mr. Shuman's question, the term "yield-point elongation" used in the paper is defined as the total elongation to the end of the yield point as recorded on an autographic curve. The automatic load-extension curves were obtained with a Kenyon-Burns wedge extensometer giving a strain magnification of ten. While the elongation at the yield point is taking place, the load is equal or less than that at the initial break in the curve.

We agree with Mr. Rupp's suggestion that a satisfactory test for evaluating the drawing and forming qualities of non-ferrous materials would be very useful. Since the paper was presented, a few tests have been made on aluminum alloys in the cup-drawing equipment with a flat-bottom punch having a $\frac{1}{8}$-in. radius. The results given below seem to agree with general opinions of the relative drawability of the materials.

Alloy	Cup-Drawing Index, per cent	Remarks
2SO	10–0	Best cup-drawing performance
52SO	9–0	
24SO	7–90	
61SO	7–20	
3S½H	6–40	
61SW	6–10	Tested 19 days after heat treatment
2S½H	6–05	Poorest cup-drawing performance

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[6] Inspecting Engineer, Jones & Laughlin Steel Corp., Pittsburgh, Pa.

[7] Chief Testing Engineer, Reynolds Alloys Co., Listerhill, Ala.

[8] Assistant Supervisor, Battelle Memorial Institute, Columbus, Ohio.

THE STRAIN AGING OF KILLED LOW-CARBON STEEL, WITH PARTICULAR REFERENCE TO THE EFFECT OF TITANIUM

BY GEORGE F. COMSTOCK[1]

SYNOPSIS

The strain aging of heat-treated specimens cut from forged bars of killed low-carbon steel melted in a small induction furnace was studied by six methods, including comparison of the yield point, and noting the changes in impact value, hardness, and damping capacity on aging after straining. Results by the damping method were found to agree with those by the yield point method, but the impact and hardness methods did not classify different steels in the same way. Aluminum deoxidation of these steels was effective in preventing the kind of strain aging shown by impact tests, but its influence on the kind shown by yield point tests was slight. The effect of nitrogen was negligible in these killed steels. Titanium if present to the extent of at least 4.5 times the carbon content was effective in preventing strain aging as determined by all the methods. Such steel does not have a real yield point whether normalized, annealed, or strained and aged.

Strain aging in this paper is considered to be the change in mechanical properties that occurs spontaneously with the passage of time, at either ordinary or elevated temperatures, after some kind of cold deformation.

The present work was prompted by an article by Hayes and Griffis (**1**)[2] in which these authors claimed that titanium, as well as aluminum, was useful for making non-aging steel. They showed that the aging tendency of low-carbon steel containing titanium was eliminated when treated by normalizing followed by at least 3 hr. annealing at about 1180 F. and slow cooling. The aging tendency was evaluated by means of the tensile stress-strain curve after straining and aging, such a curve showing a marked break at the yield point in the aging steels, but being smooth in the non-aging steels, the same as directly after straining. Because of this lack of recurrence of the yield point, non-aging steel does not suffer from "stretcher strains" in cold-formed sheets, and is, therefore, desirable for many stamped or drawn parts, such as automobile fenders and washing-machine tubs.

The cause of strain aging in low-carbon steel has been variously ascribed to oxygen (**2**, **4**, **8**), or to nitrogen (**3**, **7**), or to carbon (**11**), and no general agreement on this question has yet been reached. In American practice so-called non-aging steel has been made in large tonnages by the use of aluminum (**4**, **8**) and careful heat-treatment (**1**), but the product has not always been entirely satisfactory. The problem is still admittedly complex (**9**), aluminum deoxidation not being invariably effective (10, 11).

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[1] Metallurgist, The Titanium Alloy Manufacturing Co., Niagara Falls, N. Y.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 541.

One reason for disagreement as to the cause of strain aging is the use of different methods of testing. Beside the yield-point method already mentioned (1, 4, 11), loss of impact resistance (3, 6, 12), increase in hardness of a Brinell impression (13), and decrease of damping capacity (5, 14) have been recommended and used for determining aging by various investigators.

Methods of Testing:

Six different methods of testing for susceptibility to strain aging were used in this work, as follows:

1. Automatic plotting of the load-deformation diagram of a tension test, with a specimen first adequately strained, and then tested after a definite period of aging. The elongation at the yield point is an indication of the amount of aging (1, 4, 11).
2. Determining first the stress required for 5 per cent strain, and after a period of aging, determining the yield strength. The amount by which the latter exceeds the former is a measure of aging (11).
3. Determining the Izod impact resistance of a specimen strained in tension 5 per cent, first directly after straining, and secondly after a period of aging. A decrease is evidence of strain aging (3, 4, 12).
4. The work-brittleness test, involving Izod impact tests of specimens strained by cold drawing through a die (5, 6). Using a tapered specimen, the amount of strain is varied, and the aging factor may be introduced between straining and impact testing.
5. Measuring the Rockwell hardness of Brinell impressions, some immediately, and some after an aging period. The increase in hardness should be a measure of aging (13).
6. Damping records of specimens strained 4 per cent in tension, made 15 min. and 7 days, respectively, after straining. The decrease in the latter value below the former indicated the degree of aging (5, 14).

Methods 1, 2, and 6 were found generally to classify steels in about the same way regarding strain aging, while methods 3, 4, and 5 classified them quite differently. These two groups of test methods evidently determine different kinds of strain aging, which should be distinguished in any discussion of the subject.

Materials Used:

All the steels used in this work were melted in a basic-lined 17-lb. induction furnace, each heat being poured in a single ingot about 2.5 in. square. The steels ranged in analysis from 0.022 to 0.135 per cent carbon, 0.16 to 0.56 per cent manganese, 0.002 to 0.23 per cent silicon, 0.0033 to 0.0196 per cent nitrogen, and also contained about 0.015 per cent phosphorus, about 0.035 per cent sulfur, and up to 0.705 per cent titanium. Manganese and silicon were added to all the heats after melting, so that all the steel was killed and the ingots were sound. Aluminum up to 0.15 per cent was also added to many (but not all) of the heats. When high nitrogen was desired, about 0.2 per cent was added, after deoxidation, in the form of sodium cyanide. Titanium was added in the form of 40 per cent low-carbon ferrotitanium containing about 7 per cent aluminum, as would be done in commercial practice when a definite titanium content in the steel is required.

Most of the ingots were forged to $\frac{7}{8}$-in. round bars, but some were forged $\frac{17}{32}$ in. square. When heat treatment was used the bars were heated before machining. Normalizing in all this work was for 1.5 to 2 hr. at 1650 or 1700 F. followed by cooling in still air, and annealing was for 6 hr. at 1150 or 1180 F. followed by slow cooling in the furnace, the higher temperatures being used for the lower-carbon steels. Throughout this paper, the term "annealing" refers to subcritical treatments in the sense of process annealing. Most of the tension test specimens were machined about 0.45 in. in diameter with a 6-in. gage length. The impact test specimens were either 0.394 in. square or 0.45 in. round, and were notched with 45-deg. V-shaped notches according to the standard for Izod tests.

EFFECT OF ALUMINUM

Data obtained on steels made without any titanium addition are reported in Table I. Some of these steels were deoxidized merely with manganese and silicon, while in others various amounts of aluminum were also used.

TABLE I—STRAIN AGING DATA ON STEELS TREATED WITH DIFFERENT AMOUNTS OF ALUMINUM

Steel	Aluminum Added, per cent	Chemical Composition, per cent				Yield Point Elongation After Aging, per cent	Impact Values, ft-lb.				Rockwell B Hardness of Brinell Impression			Specific Damping, per cent		
		Carbon	Manganese	Silicon	Aluminum		Strained in Tension		Strained by Cold Drawing		Not Aged	Aged	Increase	Aged 15 Min.	Aged 7 Days	Decrease
							Not Aged	Aged	Not Aged	Aged						
NORMALIZED SPECIMENS STRAINED 4 PER CENT; AGED AT 450 F. FOR 1 HR.																
No. 1	0	0.07	0.16	0.11	0.001	3.1					86.5	95	8.5			
No. 2	0	0.10	0.37	0.20	0.001	1.9					88.5	96	7.5			
No. 4	0.05	0.09	0.40	0.22	0.05	2.9					87.5	93	5.5			
No. 6	0.10	0.10	0.42	0.18	0.095	2.2					88	92.5	4.5			
No. 8	0.15	0.09	0.35	0.12	0.14	3.0					83.5	91	7.5			
NORMALIZED SPECIMENS STRAINED 5 TO 7 PER CENT; AGED AT ROOM TEMPERATURE FOR 3 WEEKS																
No. 3	0	0.09	0.52	0.19					125.5	116.5						
No. 5	0.05	0.05	0.36	0.09		1.1			135	137						
No. 7	0.10	0.06	0.42	0.11		1.1			135	130						
No. 9	0.15	0.04	0.31	0.07		1.1			130	137.5						
NORMALIZED AND ANNEALED SPECIMENS STRAINED 5 TO 8 PER CENT; AGED AT 450 F. FOR 1 HR.																
No. 1	0	0.07	0.16	0.11	0.001	2.5	88	98			85	88.5	3.5			
No. 2	0	0.10	0.37	0.20	0.001	2.5	115	114			83.5	90.5	7			
No. 3	0	0.09	0.52	0.19		1.7					83	90.5	7.5			
No. 4	0.05	0.09	0.40	0.22	0.05	1.1	115	123			86	87	1			
No. 6	0.10	0.10	0.42	0.18	0.095	1.1	116	123			85	87.5	2.5			
No. 8	0.15	0.09	0.35	0.12	0.14	1.5	120	123			83.5	81.5	...			
NORMALIZED AND ANNEALED SPECIMENS STRAINED 4 TO 7 PER CENT; AGED AT ROOM TEMPERATURE FOR 3 WEEKS[a]																
No. 1	0	0.07	0.16	0.11	0.001	1.6	94	38			85.5	91.5	6			
No. 2	0	0.10	0.37	0.20	0.001	1.5	117	120			78	85	7	6.5	1.4	5.15
No. 3	0	0.09	0.52	0.19		1.45			132	135	85.3	83.5	...			
No. 4	0.05	0.09	0.40	0.22	0.05	0.45	127	123			84	83.5	...	7.5	2.9	4.6
No. 5	0.05	0.05	0.36	0.09		1.5	134	138	136	138.5				7.8	1.6	6.2
No. 6	0.10	0.10	0.42	0.18	0.095	0.6	116	123			85.5	82	...	7.7	3.7	4.0
No. 7	0.10	0.06	0.42	0.11		1.4	138	140	130.5	139				8.1	1.5	6.6
No. 8	0.15	0.09	0.35	0.12	0.14	0.8	120	123			84.5	84.5	...	7.75	2.8	4.95
No. 9	0.15	0.04	0.31	0.07		1.6	136	137	125.5	135				8.0	1.4	6.6

[a] Aging for the damping tests was for only one week.

The results reported in Table I show that all of the aluminum-treated steels had a well-marked yield point, whether normalized or annealed, in the strained condition after aging either 3 weeks at room temperature or for 1 hr. at 450 F. Thus aluminum was not found to be effective in eliminating this kind of strain aging. In the higher-carbon steels, however, such as Nos. 4, 6, and 8, when annealed, strained, and aged at room temperature, the yield point was definitely less well marked when aluminum deoxidation was used than in similar silicon-deoxidized steels such as Nos. 2 and 3; and it was also less well marked in the higher-carbon steels than in lower-carbon steels similarly treated with aluminum. Thus under the particular conditions of carbon content above 0.08 per cent, annealing at about 1150 F., and aging for 3 weeks at room temperature, aluminum deoxidation was found to reduce the yield point elongation, although not to prevent entirely the return of the yield point on aging after straining. It may be merely the *rate* of this kind of aging that is reduced by the aluminum. The damping capacity tests likewise reveal a slight but appreciable effect of the same nature due apparently to the differences in carbon content.

TABLE II.—STRAIN AGING DATA ON ALUMINUM-DEOXIDIZED STEELS WITH DIFFERENT NITROGEN CONTENTS.

Steel	Aluminum Added, per cent	Chemical Composition, per cent						Yield Point Elongation After Aging, per cent		Impact Values, ft-lb.						Specific Damping, per cent, Normalized and Annealed		
										Strained in Tension Normalized and Annealed		Strained by Cold Drawing						
												Normalized Only		Normalized and Annealed				
						Nitrogen												
		Carbon	Manganese	Silicon	Titanium	Soluble	Total	Normalized Only	Normalized and Annealed	Not Aged	Aged	Not Aged	Aged	Not Aged	Aged	Aged 15 min.	Aged 7 days	Decrease
SPECIMENS STRAINED 4 TO 7 PER CENT; AGED AT ROOM TEMPERATURE FOR 3 WEEKS[a]																		
Steels with no titanium:																		
No. 7....	0.10	0.06	0.42	0.11	.....	0.0040	0.0040	1.2	1.4	138	140	135	130	131	131	8.1	1.5	6.6
No. 5....	0.05	0.05	0.36	0.09	.....	0.0066	0.0066	1.1	1.5	134	138	135	137	136	138	7.8	1.6	6.2
No. 10....	0.05	0.06	0.42	0.10	.....	0.0145	0.0149	1.3	1.3	137	142	141	143	133	142	6.5	1.2	5.3
No. 11....	0.10	0.05	0.40	0.11	.....	0.0149	0.0153	1.3	1.5	140	145	142	143	134	142	7.2	1.4	5.8
Steels with low titanium:																		
No. 12....	0.05	0.04	0.36	0.09	0.068	0.0029	0.0033	1.6	1.7	141	140	139	137	131	135	7.4	1.9	5.5
No. 13....	0.15	0.05	0.32	0.07	0.090	none	0.0062	1.8	1.6	142	142	140	139	142	149	7.7	1.9	5.8
No. 14....	0.10	0.05	0.43	0.15	0.108	0.0011	0.0069	1.6	1.5	143	147	147	145	144	153	6.3	1.2	5.1
No. 15....	0.05	0.12	0.43	0.22	0.069	0.0049	0.0087	1.2	0	131	123					7.0	4.3	2.7
No. 16....	0.10	0.05	0.42	0.14	0.112	0.0049	0.0122	1.6	1.3	135	140	136	145	140	141	6.7	1.6	5.1
No. 17....	0.15	0.06	0.38	0.10	0.100	0.0008	0.0141	1.6	1.4	142	145	143	147	138	145	6.9	1.4	5.5
No. 18....	0.05	0.06	0.50	0.17	0.045	0.0090	0.0196	1.6	1.1	140	145	140	138	150	143	5.3	1.8	3.5
Steels with higher titanium:																		
No. 19....	0.05	0.07	0.41	0.19	0.213	0.0011	0.0045	2.0	1.7	143	153	150	151	155	157	5.2	1.2	4.0
No. 20....	0.10	0.10	0.40	0.20	0.188	0.0046	0.0107		0.6			149	150	152	155			
No. 21....	0.05	0.06	0.49	0.20	0.202	0.0021	0.0176		1.4			153	153	158	160			
SPECIMENS STRAINED 5 TO 8 PER CENT, AGED AT 450 F. FOR 1 HR.																		
No. 15....	0.05	0.12	0.43	0.22	0.069	0.0049	0.0087	2.1	1.3	113	125							
No. 20....	0.10	0.10	0.40	0.20	0.188	0.0046	0.0107		1.3									
No. 21....	0.05	0.06	0.49	0.20	0.202	0.0021	0.0176		1.4									

[a] Aging for the damping tests was only for one week.

With the impact and hardness tests reported in Table I, the steels deoxidized with aluminum show, with few exceptions, less aging after straining than those less thoroughly deoxidized. The improvement due to aluminum is especially well marked with the annealed specimens. This indicates some basis for the reports in the literature that strain aging is prevented by strong deoxidation with aluminum. Such a conclusion should be restricted, however, to the kind of strain aging indicated by impact and possibly hardness tests, and is not applicable except in a very limited and incomplete manner to the kind revealed either by damping tests or by the nature of the yield point, at least in these steels made on a laboratory scale and tested in bar form.

EFFECT OF NITROGEN

Some evidence on the effect of nitrogen can be derived from the data in Table I, since steel No. 1 was found to contain 0.012 per cent, steel No. 2 to contain 0.008 per cent, and steel No. 3 to contain 0.011 per cent nitrogen. No correlation can be found between these values and the strain aging data in the table, although there is some slight correlation between the data and the manganese and silicon contents. This seems to indicate that deoxidation has more influence on strain aging than nitrogen has.

Most of the data on the effect of nitrogen applies to steels deoxidized with aluminum and containing various amounts of titanium. These data are reported in Table II, where they are grouped in such a way as to minimize the effect of variations in titanium content.

The only possible conclusion from the data in Table II is that in these killed steels nitrogen has no effect on strain aging. This applies both to the kind of aging shown by the form of the yield point and by the damping tests, as well as to the kind shown by impact tests, but it must be clearly restricted to killed steels, made on a laboratory scale, and with soluble nitrogen contents below 0.015 per cent.

All the steels in Table II are practically non-aging by impact test methods, but all but one are definitely aging by the yield point method. The exception, No. 15, shows the least aging by the damping test method, but the most by the impact test method. Although this steel displayed no yield point when tested in the annealed condition after straining and aging at room temperature, it had a very definite yield point when not annealed, or even when an annealed specimen was aged for 1 hr. at 450 F. Its high carbon content should be noted, and this is believed to be the chief reason for the apparent lack of aging of the annealed specimen at room temperature. Steel No. 20 was somewhat similar, and likewise Nos. 4 and 6 in Table I. These steels all exhibited aging at 450 F. by the yield point method, although their comparatively high carbon contents apparently caused them to age at room temperature, when annealed, much more slowly than the other steels. It may be noted that both steels Nos. 15 and 20 show intermediate nitrogen contents and were not especially low in nitrogen.

Some support is found here for the claims of Hayes and Griffis (1) that annealing at about 1180 F., as well as aluminum treatment and a small titanium content, are useful for preventing strain aging as revealed by the form of the yield point. The reason for the lack of agreement between the present results and those of other investigators (3, 7) regarding nitrogen may be merely because this work was restricted to killed steels, and mostly to steels treated with aluminum or titanium. The small scale of steel-making practice used in the present work may also have been a factor in these results.

TABLE III.—STRAIN AGING DATA ON TITANIUM STEELS STRAINED ABOUT 5 PER CENT AND AGED AT ROOM TEMPERATURE.

Steel	Aluminium Added, per cent	Chemical Composition, per cent					Ratio, Titanium to Carbon	Yield Point Elongation After Aging, per cent		Impact Value After Straining in Tension, ft-lb., Normalized and Annealed		Ferrite Grains per square millimeter in Impact Specimens	Specific Damping, per cent		
		Carbon	Manganese	Silicon	Nitrogen	Titanium		Normalized Only	Normalized and Annealed	Not Aged	Aged		Aged 15 Min.	Aged 7 Days	Decrease
No. 22....	0	0.06	0.40	0.08	0.0103	0.090	1.5	1.3	1.3	125	127	1198	7.3	1.8	5.5
No. 23....	0	0.04	0.38	0.12	0.0046	0.086	2.1	1.6	1.6	140	145	1105	6.9	1.5	5.4
No. 24....	0.10	0.06	0.47	0.13	0.0137	0.246	4.1	1.3	1.6	148	148	650	5.0	1.0	4.0
No. 25....	0	0.05	0.45	0.10	0.0108	0.210	4.2	1.8	0.5	145	130	730	6.1	1.9	4.2
No. 26....	0.10	0.05	0.47	0.17	0.0052	0.240	4.8	0	0	37	145	125	7.1	6.6	0.5
No. 27....	0	0.04	0.40	0.15	0.0054	0.210	5.2	0	0	145	145	150	7.4	6.9	0.5
No. 28....	0	0.04	0.39	0.14	0.0055	0.255	6.4	0	0	146	146	145	7.2	6.6	0.6
No. 29....	0.10	0.05	0.45	0.13	0.0060	0.330	6.6	0	0	12	19	175	6.9	5.8	1.1
No. 30....	0.10	0.05	0.46	0.17	0.0043	0.330	6.6	0	0	36	147	115	6.2	5.8	0.4
No. 31....	0	0.04	0.42	0.09	0.0099	0.300	7.5	0	0	41	36	85	7.8	6.6	1.2

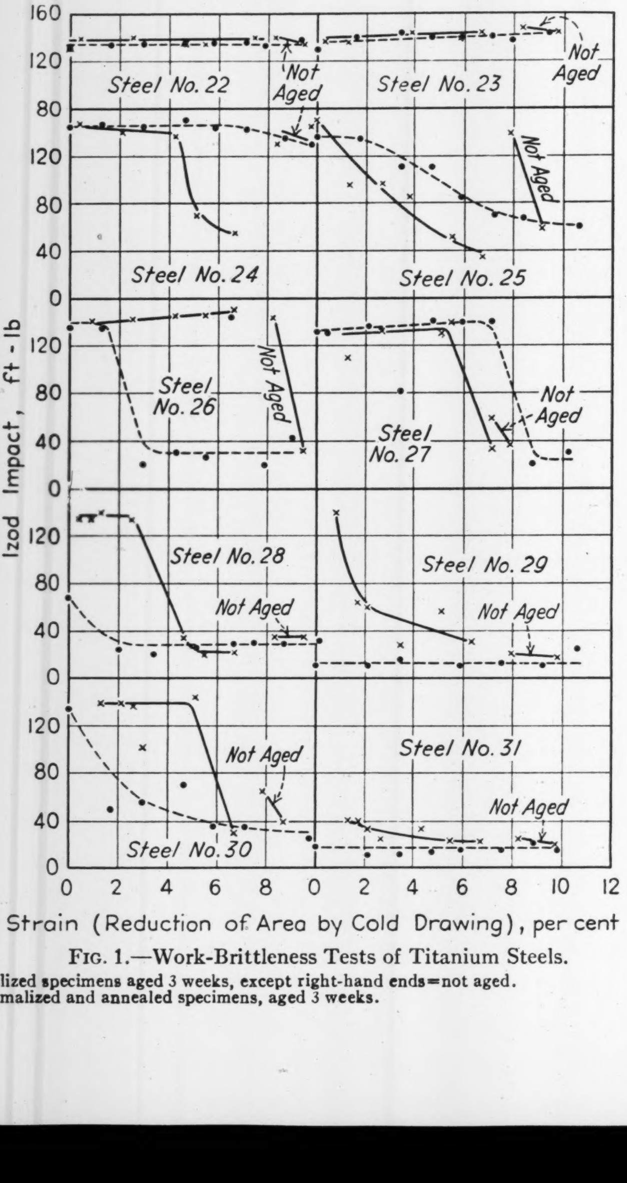

FIG. 1.—Work-Brittleness Tests of Titanium Steels.

Full lines=normalized specimens aged 3 weeks, except right-hand ends=not aged.
Dotted lines=normalized and annealed specimens, aged 3 weeks.

EFFECT OF TITANIUM ON STRAIN AGING

When this investigation was started, the chief object was to check the claim of Hayes and Griffis (1) as to the usefulness of a small titanium content in non-aging steel, and as has been noted the results from steel No. 15 seemed to support their statement in a limited fashion. In spite of many attempts, however, that steel could not be duplicated so as to show similar low aging characteristics, steel No. 20 in Table II being one of the nearest approaches although much higher in titanium.

When the English paper on strain aging (11) appeared, showing how high titanium contents in low-carbon steel would eliminate the yield point and aging, this investigation was extended to include steels of a similar nature. The conclusions of that paper were found to be well supported by our results, and with no difficulty in reproducibility. This is illustrated by the data on the higher-titanium steels in Table III. The results of work-brittleness tests (6) on these same steels are reported graphically in Fig. 1.

Table III shows very definitely that only the high-titanium steels with titanium-carbon ratios above 4.5 exhibited no yield point elongation after straining and aging, and this is true whether the steels were tested in the normalized or annealed condition, and irrespective of aluminum additions or nitrogen contents. The damping tests also showed those steels to be practically non-aging in comparison to the lower-titanium steels. These characteristics were evidently not governed by the titanium content of the steel, but by the titanium-carbon ratio; and whether this ratio was very low or just below 4.5 made little difference. The change in aging susceptibility was thus not a gradual change in the series of steels but occurred sharply as the titanium-carbon ratio rose to 4.8 or higher.

None of these steels showed appreciable aging by the impact test method, but some (not all) of the high-titanium steels gave low impact values. Steels Nos. 26 and 30 gave these low values only when not aged, and had excellent impact resistance after aging; steels Nos. 29 and 31, however, gave no high impact values, and it is interesting to note that these two showed slightly more aging by the damping test than the other four high-titanium steels. The ferrite grain sizes of the specimens were determined to throw some light on the variations in impact results, and all the high-titanium steels were found to be coarser grained than the low-titanium steels that had distinct yield points. Their impact values, however, could not be correlated with the grain size; the low impact value of steel No. 29 as compared with steels Nos. 27 and 28, for instance, is not accounted for on the basis of coarser grain size. Each impact result is the average from at least two tests which agreed closely, and no adequate explanation was found for the variations in these results.

The work-brittleness tests reported on Fig. 1 show rather irregular results. Steels Nos. 24 and 25, with titanium-carbon ratios just below the critical value, and possibly No. 30, are the only steels that seem to exhibit aging by this test. These curves prove that many steels may give good impact values and apparently no aging after only 2 per cent strain, while the results may be entirely different by the impact method of testing for strain aging if 5 per cent or more strain is applied.

The results reported in Table III were checked by slightly different tests on a few additional steels of similar nature, and these later results are shown in Table IV. In this work straining only 2 per cent and aging 16 hr. at 200 C. was tried, as has been specified (12) for determining strain aging, but was found to be definitely unsatisfactory, not only for straining by cold drawing as has been noted but also for tensile straining. The entire gage length of many tension test specimens was not deformed when the strain was restricted to only 2 per cent elongation, so that part of it remained in the unstrained condition and would, therefore, naturally show a yield point after aging even if the steel were actually of non-aging character. Such a test method with only 2 per cent strain is, therefore, inadequate to demonstrate conclusively whether a given steel is susceptible to any kind of strain aging, although in exaggerated cases it may be of some use.

TABLE IV.—STRAIN AGING DATA ON NORMALIZED AND ANNEALED TITANIUM STEELS.

Steel	Chemical Composition, per cent			Ratio, Titanium to Carbon	Yield Point Elongation After Straining 5 per cent and Aging, per cent			Increase in Yield Strength Due to Aging After Straining 5 per cent, per cent			Impact Specimens Strained by Cold Drawing				Ferrite Grains per sq. mm.	Rockwell B Hardness of Brinell Impression					
											Izod Value, ft-lb.										
											2 per cent Strain		7.5 per cent Strain								
	Carbon	Manganese	Silicon		Aged 1 hr. at 450 F.	Aged 16 hr. at 200 C.	Aged 3 weeks at 70 F.	Aged 1 hr at 450 F.	Aged 16 hr. at 200 C.	Aged 3 weeks at 70 F.	Not Aged	Age 16 hr. at 200 C.	Not Aged	Aged 3 weeks at 70 F.		Not Aged	Aged 1 hr. at 450 F.	Increase	Not Aged	Aged 3 weeks at 70 F.	Increase
No. 32....	0.07	0.27	0.004	0.5	1.85	1.7	1.2	20.6	23.4	20.0	135	120	124	15	975	82	88.7	6.7	75	84.3	9.3
No. 33....	0.05	0.43	0.13	2.0	1.25	1.3	1.55	12.2	9.8	9.5	145	154	148	152	1960	74.7	83.7	9.0	82.3	83	0.7
No. 34....	0.04	0.40	0.017	5.3	0	0	0	0	0	1.5	135	141	144	145	90	73	75	2.0	72.7	73	0.3
No. 35....	0.05	0.44	0.10	5.5	0.2	0.2	0	0.2	1.4	0.9	12.5	12.5	19	10	85	77.3	79.3	2.0	74.7	78.3	3.6
No. 36 ...	0.03	0.46	0.12	5.8	0	0	0	0	0	1.9	91	100	150	150	420	73.7	78.3	4.6	76.3	78	1.7
No. 37....	0.05	0.43	0.14	6.4	0	0	0	0	1.5	1.9	12.5	13.5	10	8	155	79.3	73.5	...	85.3	81.3	...

The results in Table IV support those previously reported in indicating practically non-aging quality in those steels having a titanium-carbon ratio above 5. Steel No. 35 seems in a slight degree exceptional, but the 0.2 per cent yield point elongation found on aging at high temperature was barely appreciable and may perhaps be considered as within the experimental error. Less than 2 per cent increase in yield strength due to aging is also probably negligible and within the experimental error. Steel No. 32 shows considerable strain-aging by every method, in spite of a small titanium content, but its low manganese and silicon content indicate that it was probably more highly oxidized in melting than the other steels. The comparison of this steel with the others in Table IV furnishes additional support to the conclusion in the section on Effect of Aluminum that deoxidation is important in preventing strain aging of the kind revealed by impact and hardness tests, but steel no. 33 shows that it is not sufficient for preventing the kind of strain-aging shown by the yield point methods.

EFFECT OF TITANIUM ON THE TENSILE PROPERTIES

When the investigation had reached this stage, the question arose as to whether titanium above 4.5 times the carbon content would not eliminate the yield point of steel even if it were not heat treated or strained, and also if the carbon content were somewhat higher than in the titanium steels tested so far. Another point of interest that remained to be cleared up was the effect of soaking the ingots at a high temperature, as would be done in large-scale commercial practice, on the yield point and aging properties of the high-titanium steels.

Therefore, two additional series of titanium steels were made, both series including steels of 0.12 to 0.135 per cent carbon as well as of lower carbon. One series of ingots was heated as usual to about 1750 F. before forging, and the other series was soaked for several hours at about 2150 F. according to usual soaking-pit practice. The analyses of these steels are given in Table V. The aluminum additions vary because all the ingots were not made at the same time, some of them having been left over from previous work; but in titanium steels, as has already been shown, such variations in aluminum treatment are of minor importance as far as the properties of the steel are concerned and may safely be disregarded here. Aluminum deoxidation is generally advisable for titanium alloy steels to increase the recovery of titanium, the aluminum being added first.

TABLE V.—CHEMICAL ANALYSES OF THE INGOTS USED.

Heated to About 1750 F. Before Forging							Soaked at About 2150 F. Before Forging						
Steel	Aluminum Added, per cent	Carbon, per cent	Manganese, per cent	Silicon, per cent	Titanium, per cent	Ratio, Titanium to Carbon	Steel	Aluminum Added, per cent	Carbon, per cent	Manganese, per cent	Silicon, per cent	Titanium, per cent	Ratio, Titanium to Carbon
No. 38.....	0.10	0.035	0.42	0.15	none	...	No. 42....	0.10	0.037	0.42	0.13	none	...
No. 39.....	0.05	0.032	0.38	0.002	0.033	1.0	No. 43....	0.05	0.022	0.34	none	0.032	1.4
No. 40.....	none	0.040	0.20	0.07	0.090	2.2	No. 44....	0.10	0.042	0.15	0.016	0.090	2.1
No. 41.....	0.05	0.044	0.30	0.07	0.195	4.4	No. 45....	0.05	0.041	0.35	0.12	0.207	5.0
							No. 50....	none	0.124	0.43	0.19	none	...
No. 46.....	0.05	0.134	0.56	0.14	0.059	0.4	No. 51....	0.10	0.126	0.38	0.18	0.042	0.3
No. 47.....	none	0.120	0.42	0.23	0.202	1.7	No. 52....	0.10	0.130	0.38	0.19	0.210	1.6
No. 48.....	0.05	0.131	0.51	0.20	0.495	3.8	No. 53....	0.05	0.131	0.44	0.13	0.504	3.8
No. 49.....	0.05	0.124	0.46	0.17	0.615	5.0	No. 54....	0.05	0.135	0.50	0.20	0.705	5.2

The ingots listed in Table V were forged to $\frac{7}{8}$-in. round bars, and all of them were finished at about the same temperature. Only tension test specimens were made from these bars, and they were all of the same form and tested in the same way as has been described. The two specimens from the upper part of each ingot, below the sinkhead, were tested in the as-forged condition, without heat treatment. One was not strained before testing, while the other was strained to 5 per cent elongation and then aged before testing. The aging of the lower-carbon steels, Nos 38 to 45 inclusive, was for 3 weeks at room temperature, while steels Nos. 46 to 54 containing 0.12 to 0.135 per cent carbon were aged at room temperature for 3 weeks and also at 450 F. for 1 hr. before testing.

The results recorded for the as-forged test specimens are given in Tables VI and VII, and most of the load-deformation curves are reproduced in Fig. 2. Those omitted were all from strained and aged bars, and were more or less inaccurate either because the specimen broke outside the points where the strainometer was attached, or because the load scale used for the diagram had to be different because of excessive strength, so that the precision of the curves was poor. All the ductility

TABLE VI.—TENSION TESTS OF AS-FORGED SPECIMENS.

Steel	Ratio, Titanium to Carbon	Yield Strength (from Fig. 2), psi.	Yield Point by Drop of Beam, psi.	Tensile Strength, psi.	Elongation, per cent		Reduction of Area, per cent
					At Yield Point in 4.5 in.	Total in 6 in.	
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS NOT SUPERHEATED							
No. 38	0	42 000	42 800	51 700	5.35	24.5	80.5
No. 39	1.0	39 000	40 800	50 300	3.1	25.0	79.8
No. 40	2.2	44 800	44 800	52 100	4.75	25.8	77.1
No. 41	4.4	36 100	.....	52 500	0	20.5	87.0
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS SUPERHEATED							
No. 42	0	40 000	40 000	54 000	1.65	19.5	74.8
No. 43	1.4	40 200	40 200	51 600	1.35	17.8	69.9
No. 44	2.1	44 000	44 000	55 100	6.0	13.3	78.3
No. 45	5.0	41 700	.....	56 700	0	15.0	87.0
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS NOT SUPERHEATED							
No. 46	0.4	53 000	54 600	71 000	3.1	23.0	65.2
No. 47	1.7	58 600	58 600	73 300	3.3	19.7	72.2
No. 48	3.8	57 000	59 700	69 400	3.8+	17.0	79.1
No. 49	5.0	28 500	.....	60 000	0	21.0	69.9
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS SUPERHEATED							
No. 50	0	51 800	52 500	65 200	1.55	16.3	66.0
No. 51	0.3	54 000	54 000	67 000	2.15	13.2	69.8
No. 52	1.6	57 500	61 500	75 100	1.2	14.7	73.6
No. 53	3.8	54 200	60 000	70 000	1.1	12.0	79.1
No 54	5.2	51 100	.....	64 900	0	11.8	77.9

TABLE VII.—TENSION TESTS OF AS-FORGED SPECIMENS STRAINED AND AGED.

Steel	Ratio, Titanium to Carbon	Yield Strength Before Straining, psi.	Stress for 5 per cent Strain, psi.	After Straining and Aging			Elongation, per cent		Reduction of Area, per cent
				Yield Strength (from Fig. 2), psi.	Drop of Beam, psi.	Tensile Strength, psi.	At Yield Point in 4.5 in.	Total in 6 in.	
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS NOT SUPERHEATED									
No. 38	0	46 500	46 500	47 100	54 800	57 400	2.3	16.3	78.9
No. 39	1.0	39 500	43 100	53 500	54 000	59 700	2.1	19.7	73.5
No. 40	2.2	46 500	47 800	55 000	55 500	55 800	5.3	22.0	77.8
No. 41	4.4	34 300?	47 200	50 100	.....	55 100	0	27.8	87.5
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS SUPERHEATED									
No. 42	0	40 000	51 000	58 000	58 600	58 600	2.7+	13.3	74.2
No. 43	1.4	38 800	48 400	57 000	57 800	60 000	2.6	15.2	78.5
No. 44	2.1	52 800	59 600	?	64 600	64 600	?	10.3	77.6
No. 45	5.0	49 800?	57 900	57 800	61 600?	62 100	0	14.7	84.3
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS NOT SUPERHEATED									
No. 46	0.4	53 800	61 000	72 300	74 200	80 500	3.1	20.2	62.3
No. 47	1.7	59 900	64 300	73 000	73 500	77 600	2.45	15.3	68.6
No. 48	3.8	61 500	62 800	?	77 400	80 000	3.3	16.7	77.7
No. 49	5.0	29 400?	53 500	53 200	56 000	62 700	0.35?	24.2	81.2
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS SUPERHEATED									
No. 50	0	58 500	63 700	?	76 300	78 100	3.3	15.5	62.1
No. 51	0.3	59 000	64 500	72 300	74 300	74 300	?	10.0	66.7
No. 52	1.6	48 800	49 900	62 100	62 500	67 500	2.4	22.0	70.0
No. 53	3.8	58 300	67 900	?	81 500	81 500	?	13.3	76.5
No. 54	5.2	52 000?	64 000	60 200	67 300?	68 500	0.55	15.8	76.8

values in the tables are given as per cent of the original length or area, except for the reductions of area of the strained and aged specimens which, like the stress values of the same specimens, were calculated on the basis of the cross-sectional area after straining.

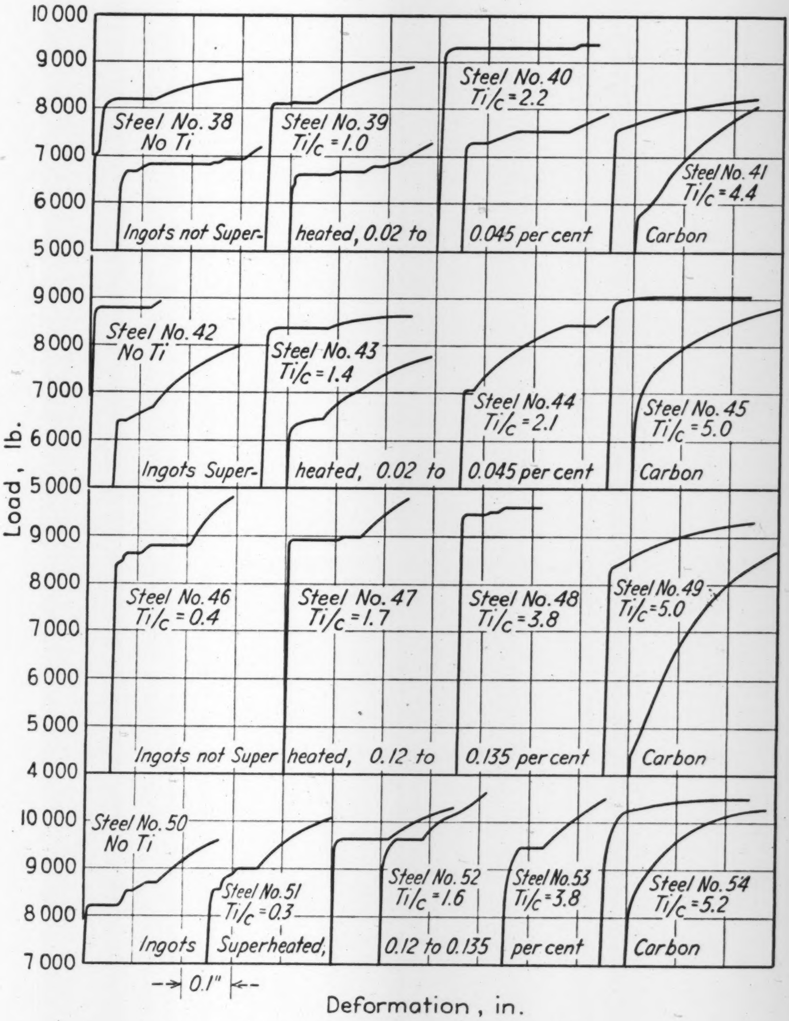

FIG. 2.—Load-Deformation Diagrams of As-Forged Specimens.

Where two curves are shown for the same steel, the upper left one represents a strained and aged specimen. The other curves represent specimens without previous straining.

The two specimens from the middle portion of each bar were normalized for 1.5 hr. at 1700 F. and then tested in the same way as has been described for the as-forged specimens, one without preliminary straining and the other after straining and aging in the stated man-

TABLE VIII.—TENSION TESTS OF NORMALIZED SPECIMENS.

Steel	Ratio, Titanium to Carbon	Yield Strength (from Fig. 3), psi.	Yield Point by Drop of Beam, psi.	Tensile Strength, psi.	Elongation, per cent		Reduction of Area, per cent
					At Yield Point in 4.5 in.	Total in 6 in.	
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS NOT SUPERHEATED							
No. 38	0	35 200	36 300	50 000	4.45	28.0	78.9
No. 39	1.0	33 400	36 100	49 400	4.65	33.3	81.6
No. 40	2.2	34 000	34 100	48 600	4.35	29.2	79.4
No. 41	4.4	33 400	.....	48 900	0	9.7	86.1
STEELS WITH 0.02 TO 0.045 PER CENT CARBON INGOTS SUPERHEATED							
No. 42	0	33 000	33 100	49 100	4.45	31.7	79.2
No. 43	1.4	26 200	28 400	46 900	2.7	33.3	85.1
No. 44	2.1	34 000	34 100	48 200	4.05	31.7	79.2
No. 45	5.0	15 500	.....	45 000	0	27.5	88.7
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS NOT SUPERHEATED							
No. 46	0.4	51 000	51 000	69 600	3.9	25.0	69.0
No. 47	1.7	46 000	46 400	59 900	4.4	27.0	73.0
No. 48	3.8	48 200	48 900	66 000	3.5	22.0	81.4
No. 49	5.0	21 000	.....	56 300	0	23.2	79.6
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS SUPERHEATED							
No. 50	0	38 000	38 900	58 800	3.25	28.7	68.1
No. 51	0.3	39 800	40 100	56 200	4.35	28.5	73.0
No. 52	1.6	44 700	44 800	59 400	4.55	26.7	73.1
No. 53	3.8	35 200	35 200	58 000	2.1	24.0	83.9
No. 54	5.2	20 400	.....	53 000	0	25.7	83.0

TABLE IX.—TENSION TESTS OF NORMALIZED SPECIMENS STRAINED AND AGED.

Steel	Ratio, Titanium to Carbon	Yield Strength Before Straining, psi.	Stress for 5 per cent Strain, psi.	After Straining and Aging			Elongation, per cent		Reduction of Area, per cent
				Yield Strength (from Fig. 3), psi.	Drop of Beam, psi.	Tensile Strength, psi.	At Yield Point in 4.5 in.	Total in 6 in.	
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS NOT SUPERHEATED									
No. 38	0	35 400	40 000	47 800	48 600	57 500	1.4	21.7	77.5
No. 39	1.0	36 600	39 000	48 500	49 600	60 000	1.65	23.7	78.1
No. 40	2.2	34 800	39 600	47 800	49 100	56 300	1.6	25.8	76.8
No. 41	4.4	22 300?	38 600	40 400	41 300	48 400	0.55	27.7	88.9
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS SUPERHEATED									
No. 42	0	36 400	40 000	47 800	48 600	57 600	1.4	24.0	75.0
No. 43	1.4	28 600	36 200	44 800	46 100	57 000	1.65	25.2	81.2
No. 44	2.1	32 500	38 800	46 000	47 300	54 900	1.55	20.8	75.0
No. 45	5.0	21 100?	36 900	39 100	.....	47 000	0	32.2	88.9
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS NOT SUPERHEATED									
No. 46	0.4	52 100	58 900	71 400	73 500	79 500	3.05	20.5	62.2
No. 47	1.7	45 600	49 700	61 400	62 000	68 000	2.7	20.8	72.9
No. 48	3.8	50 600	58 400	69 800	70 700	79 800	2.3	18.2	78.3
No. 49	5.0	23 600?	48 100	50 600	.....	57 800	0	21.3	81.0
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS SUPERHEATED									
No. 50	0	40 100	48 300	59 200	59 800	69 100	2.1	23.3	65.0
No. 51	0.3	42 800	46 200	53 500	55 000	62 100	1.75	24.0	70.2
No. 52	1.6	46 000	50 100	60 000	60 500	67 000	2.0	22.7	69.8
No. 53	3.8	39 500	50 700	59 000	59 900	73 100	2.05	20.3	79.5
No. 54	5.2	19 800?	44 000	46 000	.....	55 000	0	25.8	81.2

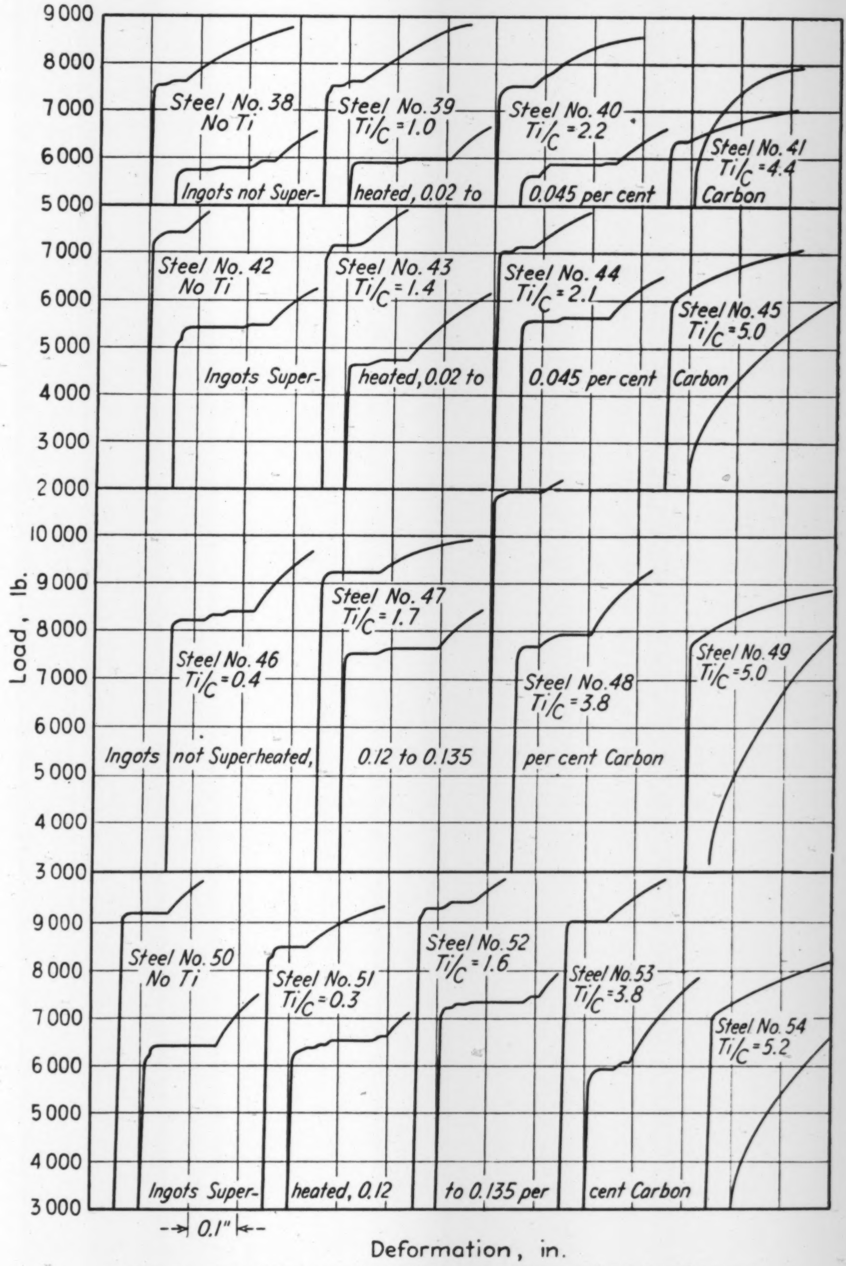

FIG. 3.—Load-Deformation Diagrams of Normalized Specimens.

The left curve of each pair represents a strained and aged specimen. The curve to the right, in each pair, represents a specimen not strained before testing.

TABLE X.—TENSION TESTS OF NORMALIZED AND ANNEALED SPECIMENS.

Steel	Ratio, Titanium to Carbon	Yield Strength (from Fig. 4), psi.	Yield Point by Drop of Beam, psi.	Tensile Strength, psi.	Elongation, per cent		Reduction of Area, per cent
					At Yield Point in 4.5 in.	Total in 6 in.	
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS NOT SUPERHEATED							
No. 38....	0	30 500	30 500	46 300	3.9	32.0	84.3
No. 39....	1.0	30 600	31 900	46 800	4.3	32.2	83.0
No. 40....	2.2	31 000	31 300	45 800	3.2	32.2	82.6
No. 41....	4.4	16 300	.....	44 700	0	28.0	89.0
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS SUPERHEATED							
No. 42....	0	30 500	31 000	46 400	3.7	33.7	82.6
No. 43....	1.4	24 300	26 600	44 100	3.0	31.3	84.3
No. 44....	2.1	26 700	27 300	44 100	2.85	32.3	83.1
No. 45....	5.0	15 600	.....	45 000	0	32.8	89.4
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS NOT SUPERHEATED							
No. 46....	0.4	45 500	46 100	60 600	4.3	26.7	75.0
No. 47....	1.7	41 000	41 600	57 900	4.05	26.7	77.5
No. 48....	3.8	44 900	44 900	63 300	3.3	23.3	81.4
No. 49....	5.0	21 000	.....	54 800	0	20.8	77.8
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS SUPERHEATED							
No. 50....	0	33 000	33 800	52 500	3.4	28.3	73.2
No. 51....	0.3	35 400	36 000	59 600	4.25	32.7	75.1
No. 52....	1.6	40 500	40 600	57 200	3.35	28.3	77.5
No. 53....	3.8	31 500	32 900	55 800	1.25	28.0	84.4
No. 54....	5.2	16 700	.....	52 500	0	25.8	80.7

TABLE XI.—TENSION TESTS OF NORMALIZED AND ANNEALED SPECIMENS, STRAINED AND AGED.

Steel	Ratio, Titanium to Carbon	Yield Strength Before Straining, psi.	Stress for 5 per cent Strain, psi.	After Straining and Aging			Elongation, per cent		Reduction of Area, per cent
				Yield Strength (from Fig. 4), psi.	Drop of Beam, psi.	Tensile Strength, psi.	At Yield Point in 4.5 in.	Total in 6 in.	
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS NOT SUPERHEATED									
No. 38.............	0	33 200	37 300	42 800	43 500	49 100	1.4	33.3	82.6
No. 39.............	1.0	31 600	37 200	45 900	46 300	53 100	1.8	27.0	81.1
No. 40.............	2.2	31 400	37 600	43 500	44 300	48 800	2.0	34.3	80.7
No. 41.............	4.4	18 600?	38 300	40 100	.....	47 500	0	32.5	87.4
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS SUPERHEATED									
No. 42.............	0	32 500	37 400	42 800	43 700	49 700	1.6	33.2	79.9
No. 43.............	1.4	28 000	34 700	43 100	43 600	52 500	1.65	24.3	80.5
No. 44.............	2.1	29 000	35 800	41 300	42 000	47 600	2.2	31.7	79.8
No. 45.............	5.0	18 600?	37 600	38 500	.....	47 800	0	32.5	88.2
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS NOT SUPERHEATED									
No. 46.............	0.4	47 400	52 700	59 300	59 700	64 800	1.55	22.3	67.6
No. 47.............	1.7	42 500	50 200	56 700	57 500	63 300	1.5	25.7	75.0
No. 48.............	3.8	46 700	56 300	65 400	66 000	71 100	1.8	17.2	79.0
No. 49.............	5.0	22 200?	48 500	50 500	.....	58 000	0	18.8	77.5
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS SUPERHEATED									
No. 50.............	0	33 800	42 800	51 500	52 200	58 000	1.95	20.3	71.2
No. 51.............	0.3	38 400	43 800	48 600	49 500	56 600	1.0	31.0	74.2
No. 52.............	1.6	41 900	48 200	54 000	54 600	61 000	1.3	25.7	74.4
No. 53.............	3.8	33 800	49 000	57 700	58 500	64 900	1.75	19.3	80.6
No. 54.............	5.2	18 600?	43 800	46 000	.....	55 000	0	25.3	81.3

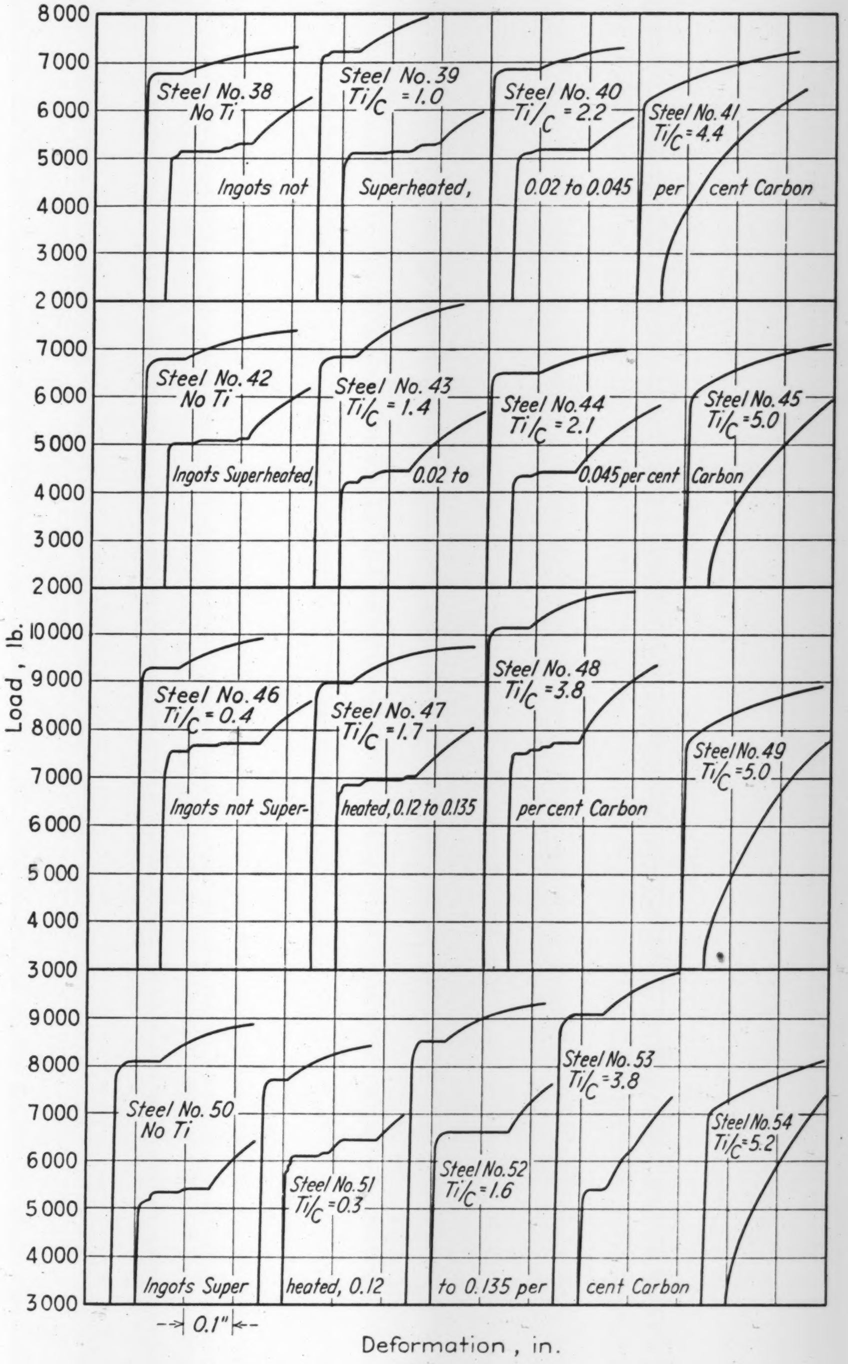

FIG. 4.—Load-Deformation Diagrams of Normalized and Annealed Specimens.

The left curve of each pair represents a strained and aged specimen. The curve to the right, in each pair, represents a specimen not strained before testing.

ner. The results are given in Tables VIII and IX, and Fig. 3.

The two specimens from the end of the forged bar made from the bottom of each ingot were normalized 1.5 hr. at 1700 F. and annealed 6 hr. at 1180 F., followed by slow cooling in the furnace. They were then machined and tested in the same way as the others, one from each ingot without preliminary straining and the other after straining and aging. The results are given in Tables X and XI, and Fig. 4.

TABLE XII.—PERCENTAGE INCREASES IN TENSILE PROPERTIES OF STEELS WITH TITANIUM-CARBON RATIOS ABOVE FOUR, AS COMPARED WITH NON-TITANIUM STEELS.

	As-Forged	As-Forged, Strained, and Aged	Normalized	Normalized, Strained, and Aged	Normalized and Annealed	Normalized, Annealed, Strained, and Aged
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS NOT SUPERHEATED						
Yield strength	−20.4	6.4	−21	−15.5	−45	−6.3
Tensile strength	1.5	−4	−2.2	−15.8	−3.5	−3.3
Elongation in 6 in.	−16	70	−65	27.6	−12.5	−2.4
Reduction of area	8.1	10.9	9.1	14.7	5.6	5.8
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS SUPERHEATED						
Yield strength	14.3	−0.3	−46.5	−18.2	−45.7	−10
Tensile strength	5	6	−8.3	−18.4	−3	−3.8
Elongation in 6 in.	−23	10.5	−13.3	34.2	−2.7	−2.1
Reduction of area	16.3	13.6	12	18.5	8.2	10.4
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS NOT SUPERHEATED[a]						
Yield strength	−45.8	−26.5	−56.7	−29.1	−53.5	−14.8
Tensile strength	−15.5	−22.1	−19.1	−27.3	−9.6	−10.5
Elongation in 6 in.	−8.7	19.8	7.2	3.9	−17.5	−15.7
Reduction of area	7.2	30.4	15.3	30.3	3.7	14.6
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS SUPERHEATED						
Yield strength	−6.5	?	−48.5	−22.3	−47.1	−10.7
Tensile strength	−0.5	−12.3	−9.9	−20.4	0	−5.2
Elongation in 6 in.	−27.6	1.9	−10.5	9.9	−8.8	24.6
Reduction of area	16.7	26.9	21.9	25	10.2	14.2

[a] Since there was no non-titanium steel in this series, the percentages are based on the values for steel No. 46, with only 0.059 per cent titanium or a titanium-carbon ratio of 0.4.

The most striking features of the data in these tables and curves are the negligible or absent yield-point elongation in all the steels with titanium-carbon ratios of 4.4 or more, and only in those steels, and consequently their practically smooth load-deformation curves, while all the other steels showed curves with definite flat portions. A few slight exceptions occurred among the high-titanium steels in the as-forged, strained and aged specimens, and in heat No. 41 with a titanium-carbon ratio of 4.4. The titanium was a little low in this steel for complete removal of the yield point, and this checks the previous conclusion that the titanium should be at least 4.5 times the carbon.

The effect of superheating the ingots, as would occur in regular soaking-pit practice, is seen only in the as-forged specimens of the high-titanium steels, the yield properties in that condition of those steels that were superheated being definitely higher than those of similar steels not superheated. This may be accounted for by assuming a stiffening effect of the titanium carbide that dissolves in the austenite at high temperatures and which does not precipitate rapidly on cooling. The effect is also shown to a smaller extent in the tensile strength and in the ductilities, in Tables VI and VII, but was entirely lost on normalizing.

An anomalous result from the normalized specimen of the high-titanium steel No. 41 should perhaps be discussed, as this is the only instance (see Fig. 3) where the unstrained curve crosses the other one. This specimen had an abnormally high yield strength and low elongation, the reason for which can only be suspected as being that the specimen may have been strained by bending and straightening when it was machined outside our plant. Perhaps the fact that the titanium-carbon ratio of this steel was just below the critical value may also have been a factor in this exceptional result.

To facilitate a comparison of the properties of the nontitanium and high-titanium steels, Table XII has been prepared as a summary of Tables VI to XI, inclusive, to show how the various properties in the various conditions of heat treatment, straining, and aging, were affected by adding titanium to the extent of 4.4 to 5.2 times the carbon content.

It is readily seen from Table XII that the yield strength was nearly always very much decreased by the high titanium additions, especially with normalized specimens where the minimum decrease was 15 per cent and the maximum 56 per cent. A few exceptions occurred with as-forged specimens, but none with normalized or annealed specimens. It should be noted that the yield strength differences reported here for specimens not strained and aged were computed from averages of the two values determined without straining, which in most instances were in good agreement.

The tensile strengths of the high-titanium steels are also generally lower than those of the plain steels, but the differences are mostly not over 10 per cent, and much less than the differences in yield strength where the specimens were not previously strained and aged. The elongation differences are rather variable, owing in some instances to slightly inaccurate machining, but in all the as-forged or normalized steels after straining and aging the elongations are higher in the high-titanium steels. The reductions of area are, without exception, higher for the high-titanium steels, the differences ranging from 3 to 30 per cent.

Thus it may be concluded that with at least 4.5 times as much titanium as carbon, steels with less than 0.045 per cent carbon, or with 0.12 to 0.135 per cent carbon, have yield strengths generally much lower than similar steels without titanium, and also have better ductility. The difference in tensile strength between the two classes of steel is generally less than the difference in yield strength, and is very much less when tests are made with specimens not previously strained and aged. Furthermore, the high-titanium steels do not have a distinct yield point whether in the as-forged, normalized, annealed, or aged condition. These characteristics should make the high-titanium steels very attractive as a material for sheets that must be formed by deep drawing, since with a low yield strength and high ductility they should be easily formed, and it should be impossible to form "stretcher-strain" markings in them if no definite yield point occurs.

Another advantage for the high-titanium steel is that annealing does not seem to be necessary. It may be noted by comparing Tables VIII and X, or Tables IX and XI, that all the properties of each class of normalized high-titanium steel compare very favorably with the properties of the corresponding non-titanium steel in the normalized *and annealed* condition. Since annealing is not required with the high-titanium steel to give it a low enough yield strength, or sufficient ductility, or to make it non-aging with respect to the yield point, this step might be omitted in processing such sheets with considerable economy.

Since the the results obtained with titanium at least 4.5 times the carbon content have been shown to be practically the same with carbon below 0.045 per cent as with carbon from 0.12 to 0.135 per cent, and to agree with results that have been reported previously with intermediate carbon contents, there seems little doubt that these conclusions apply generally to all steels with carbon contents at least up to 0.135 per cent.

So far as aging is concerned, it is obvious that since the high-titanium steels do not have a distinct yield point even as forged, the absence of change in the character of their yield points on aging after straining cannot be used as evidence of non-aging quality. The data in Tables VII, IX, and XI, however, throw some light on the aging of these steels by showing the increase in stress value between straining 5 per cent and the yield strength after straining and aging. Any such increase could only be the result of aging. Table XIII gives some comparisons based on those values, but they must be considered as only rough approximations in view of the way the tension tests were made. The properties after straining and aging were calculated on the basis of the cross-section of the specimen after it had been reduced about 5 per cent, while the stress for 5 per cent strain was calculated from the cross-section before that reduction. Hence even if there were no real strength increase from aging, the yield strength as calculated after the straining, and given in Tables VII, IX and XI, would show an apparent increase of about 5 per cent over the stress previously calculated for 5 per cent strain. The figures given in Table XIII are, therefore, all reduced 5 per cent from the results obtained from computations using the values given in Tables VII, IX and XI.

TABLE XIII.—PERCENTAGE INCREASES IN YIELD STRENGTH AFTER AGING, AS COMPARED WITH THE STRESS PRODUCING THE PREVIOUS 5 PER CENT STRAIN.

Steel	Ratio, Titanium to Carbon	As-Forged	Normalized	Normalized and Annealed
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS NOT SUPERHEATED				
No. 38	0	12.8[a]	14.4	10.2
No. 39	1.0	19.1	19.3	9.7
No. 40	2.2	10.0	15.7	10.7
No. 41	4.4	1.1	−0.3	−0.3
STEELS WITH 0.02 TO 0.045 PER CENT CARBON, INGOTS SUPERHEATED				
No. 42	0	8.7	14.4	9.4
No. 43	1.4	12.8	18.8	19.2
No. 44	2.1	3.4[a]	13.5	10.3
No. 45	5.0	−5.2	1.0	−2.6
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS NOT SUPERHEATED				
No. 46	0.4	13.5	16.2	7.5
No. 47	1.7	8.5	18.6	7.9
No. 48	3.8	18.3[a]	14.5	11.2
No. 49	5.0	−5.6	0.2	−0.9
STEELS WITH 0.12 TO 0.135 PER CENT CARBON, INGOTS SUPERHEATED				
No. 50	0	14.7[a]	17.6	15.3
No. 51	0.3	7.1	10.8	6.0
No. 52	1.6	19.4	14.7	7.0
No. 53	3.8	15.0[a]	11.4	12.7
No. 54	5.2	−10.0	−0.5	0

[a] These values were determined from the drop-of-beam yield points.

Table XIII shows that there was practically no increase in strength on aging any of the high-titanium steels, while the other steels showed from 6 to 19 per cent increase, with one exception among the as-forged specimens.

It should be remembered in connection with this evidence on aging that all the higher-carbon steels were aged 1 hr. at 450 F. as well as for 3 weeks at room temperature.

The tension test results reported above confirm the statements in the English reference (11) that titanium contents sufficient to combine with all the carbon in low-carbon steel not only prevent strain aging as shown by the form of the yield point but also eliminate the yield point of the steel. Such steel, with a titanium-carbon ratio above 4.5, seems to be a fundamentally different material from ordinary killed low-carbon steel or steel treated with small amounts of titanium or aluminum or both. In yield characteristics it resembles a non-ferrous alloy. This is true of the steel in the as-forged condition as well as after heat treatment, whether superheated before forging or not. It also applies to steels with any carbon content at least up to 0.135 per cent. All of the evidence so far acquired on these steels has been derived from bars or rods made on a small laboratory scale, however, and confirmation in large-scale commercial practice, or on steel rolled to sheet form, has not yet been established.

With those limitations understood, this work supports the conclusions of the English investigators (11) that the cause of the kind of strain aging revealed by the yield point method, as well as the cause of the yield point itself, is carbon dissolved in ferrite. Elimination of dissolved oxygen alone apparently has very little effect on that kind of strain aging, although oxygen probably is the cause of the kind shown by decrease in impact values.

CONCLUSIONS

The conclusions here given apply only to low-carbon killed steels, made on a small laboratory scale and forged into bars, with aging treatments as specified in the paper. They are subject to confirmation with sheet steel made according to usual commercial practice.

1. Strain aging shown by the return of a definite yield point in a specimen strained in tension is entirely different from strain aging shown by loss of notched-bar impact value in strained specimens held for appreciable periods of time. Steels classed as "aging" by the yield point method, were usually classed as "non-aging" by the impact method. Steels classed as non-aging by the yield point method may show comparatively low impact values.

2. Whether a given steel shows "strain aging," especially by the impact test method, depends often on the amount of strain, and 2 per cent strain either in tension or by cold drawing is generally insufficient.

3. Damping capacity tests may be used to indicate susceptibility to the same kind of strain aging as is shown by the yield point method. The Sauveur hardness test method (13) is less reliable and gives results more in agreement with the impact test method.

4. Strong deoxidation as with aluminum may decrease the amount of strain aging, of the kind shown by the yield point method, in annealed steels with over 0.08 per cent carbon aged at room temperature, but otherwise it is ineffective for preventing that kind of strain aging. It does, however, decrease effectively the kind of strain aging shown by impact tests, which is, therefore, probably due to oxygen in the steel.

5. The nitrogen content of killed low-carbon steels, up to 0.015 per cent soluble or 0.02 per cent total, does not affect to an important degree the susceptibility to either kind of strain aging.

6. Steels containing as much as 0.10 to 0.12 per cent carbon, with less than 4.5 times as much titanium, show aging by the yield point method when aged at 200 C. or 450 F., but may not show as definite aging by this method in 3 weeks at room temperature, or may age more slowly at room temperature than similar lower-carbon steels. All steels of this nature that we tested were non-aging by the impact test method.

7. Low-carbon steels containing at least 4.5 times as much titanium as carbon do not show a definite yield point after straining and aging, whether aged 3 weeks at room temperature, 16 hr. at 200 C. (390 F.), or 1 hr. at 450 F. Low-carbon steels with less titanium, even though deoxidized with aluminum, show strain aging by that method under the same conditions.

8. The high-titanium non-aging low-carbon steels were coarser grained than the others, but some of them had just as good notched-bar impact resistance as the finer-grained lower-titanium steels. Some of them, however, were low in impact resistance and the reason for the difference was not discovered.

9. Increase of yield strength on aging after 5 per cent strain is negligible in low-carbon steel containing at least 4.5 times as much titanium as carbon, which has no well-defined yield point.

10. In steels containing up to 0.135 per cent carbon, the presence of titanium to the extent of at least 4.5 times the carbon content greatly lowers the yield strength of the normalized steel, decreases the tensile strength to a smaller degree, increases the ductility, and causes the steel not to show a well-marked yield point.

11. The effects mentioned in conclusion 10 do not occur gradually with increasing titanium content, but smaller amounts of titanium below the critical amount may even give a gradually increasing yield strength with titanium, up to about 4.5 times the carbon content, when the changes in properties noted in conclusions 7, 9, and 10 occur. Thus, it is only the elimination of the last traces of carbon uncombined with titanium that is effective in producing those changes.

12. When such titanium steels are heated for a long time at high temperature, as in a soaking pit, and tested without subsequent heat treatment at lower temperatures, the yield and tensile strengths are higher and the ductility lower than when ingots are not so treated, or than after normalizing.

13. This low-carbon titanium steel, with titanium-carbon ratio of at least 4.5, should be a desirable material for deep-stamping sheets since it requires only normalizing, and not annealing, to develop high ductility with low yield strength and fair tensile strength, has no definite yield point so that "stretcher strains" could not occur, and its yield strength does not increase on aging after straining.

14. The chief cause of the kind of strain aging indicated by the return of a well-marked yield point on aging after straining, as well as of the yield point itself, is probably carbon dissolved in the ferrite.

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(13) A. Sauveur and J. L. Burns, "A Method for Studying Strain Hardening Susceptibility and Aging After Cold Work Deformation," *Metals and Alloys*, Vol. 4, No. 1, January, 1933, p. 6.

(14) J. T. Norton, "A Torsion Pendulum Instrument for Measuring Internal Friction," *The Review of Scientific Instruments*, Vol. 10, March, 1939, p. 77.

DISCUSSION

Mr. D. J. McAdam, Jr.[1]—This paper discusses the effects of strain aging after plastic deformation. In some recent experiments, described in a paper to be presented at a later session,[2] I have encountered considerable strain aging during slow tension tests. Because of strain aging during a test at 100 C., the tensile strength of ingot iron was raised to a value equal to that obtained at −100 C. A qualitatively similar effect is obtained with many non-ferrous metals even at room temperature or lower.

When results of tension tests with the same metal are plotted on a scale of equal thermodynamic efficiencies, the influence of strain aging may be revealed by an abrupt upturn of the curve above a certain temperature.

Messrs. G. V. Smith[3] **and R. F. Miller**[3] (*presented in written form*).—In this interesting paper it is stated that the yield point elongation of a specimen tested after having been adequately strained and aged is an indication of the amount of aging. In our opinion, this is true, but only as an *indication*, not as a quantitative measure, particularly when the prior strain is varied and the difference in yield point elongation is less than 1.0 per cent. Moreover, the yield point elongation is markedly influenced by grain size, the elongation being greater the finer the grain size and virtually absent in very coarse-grained steels. It would be interesting, therefore, to know the grain size of the steels, particularly those of Tables I and II.

Again, records of yield point elongation before straining and aging, after straining but before aging, and the before and after stresses, would show, for example, any change in character of the yield and whether the initial yield point elongation had been exceeded in the first straining; if it had not, the presence of yield elongation after straining and aging could not be attributed necessarily to strain aging. We are of the opinion that more reliable and definite conclusions could have been drawn from the results of more systematic measurements of different properties of a smaller number of steels, in part because the amount of aluminum or other deoxidizer which was added cannot be regarded as a safe criterion of extent of deoxidation.

The conclusion that strain aging differs according as it is revealed by the return of a yield point or by the loss of impact strength is not thought to be in accord with current conceptions. Is it not that the difference is merely a matter of degree, some properties being differently affected both as to magnitude and rate of change? The ability of titanium, when present in sufficient amount, to eliminate the yield point both before and after straining and aging is very interesting, but it seems inappropriate to infer from this alone that such a steel is non-aging (see third paragraph, page 527, of the paper); or that a steel classed as non-aging by the yield method may be classed as aging by the impact test.

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[1] Metallurgist, National Bureau of Standards, Washington, D. C.

[2] D. J. McAdam, Jr. and R. W. Mebs, "The Technical Cohesive Strength and Other Mechanical Properties of Metals at Low Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 661 (1943).

[3] Research Laboratory, United States Steel Corp. of Delaware, Kearny, N. J.

We should like to know whether the author has any explanation of why the impact values of Table III for 5 per cent tensile deformation are generally high both before and after aging, yet the values plotted in Fig. 1 for the same alloys generally show a falling off to relatively low value after a certain amount of prior deformation, frequently less than 5 per cent. Incidentally, from a comparison of Table III and Fig. 1, it appears that the values in Table III for steels Nos. 26 and 30 in the not aged and aged conditions might have been reversed. Would this be possible? Further, in the opinion of the author, would it be possible that the failure of the impact test to show aging, as shown by the various tables, could be attributed to an insufficient amount of prior deformation?

We are also unable to understand how it can be concluded that, if a steel with a titanium-carbon ratio greater than 4.5 has no yield point, carbon dissolved in the ferrite is the reason for the yield point itself, for besides combining with carbon, the titanium has changed the composition of the steel and has undoubtedly combined with N_2, O_2, etc.

MR. L. J. LARSON.[4]—I should like to ask a question in connection with the strain aging of mild steels which do not contain titanium. In the fabrication of some structures of mild steel it has been observed that after cold rolling, or considerable cold forming and subsequently welding, cracks have developed in the steel plates. Investigating these materials later, it was found that the impact values were low. Such steels have satisfactory impact values in the "as-received" condition and also after fabricating and stress relieving. I wonder whether the low impact values were due to some type of strain aging. These failures were not in the weld itself but in the plate material and could, therefore, not be caused by nitrogen in the welds. In one instance, a short cylinder, about 13 ft. in diameter, was rolled cold from plates about 3½ in. thick, after which a disk was welded inside of the cylinder at each end. After the structure was completed and standing idle in the shop, a crack occurred in the cylinder extending nearly one half of the circumference. This crack followed near the center of the cylinder except for a short distance at each end where it curved to the ends of the cylinder. In the fabrication of this structure there were, undoubtedly, high stresses due to the cold rolling and additional stresses due to the shrinkage of the weld joining the disk to the inside of the cylinder at the ends. In view of Mr. Comstock's results, is it logical to assume that the fabricating strains together with the general heating of the structure during welding produced aging and resulted in low impact values and thus caused the failure?

MESSRS. F. W. BOULGER[5] AND F. B. DAHLE[5] (*presented in written form*).—Mr. Comstock is to be congratulated upon his interesting and valuable addition to the information on strain aging of low-carbon steels. It is particularly significant that he found specific titanium-carbon ratios to be necessary to prevent changes in properties during accelerated aging treatments. Very few metallurgical phenomena act with such sharply defined limits. The fact that he used so many aging criteria to check his conclusions is also to be commended. Since his titanium-carbon ratios were much smaller than those studied by the English investigators, the steels more closely approach commercially practicable materials.

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[4] Consulting Welding Engineer, Milwaukee, Wis.

[5] Research Metallurgist and Assistant Supervisor, respectively, Battelle Memorial Institute, Columbus, Ohio.

Some titanium-bearing steel sheet has been tested in our experimental cup-drawing test equipment. The steel was made commercially by ingot additions of ferrotitanium and presumably processed by the practice ordinarily used with killed sheet. The results of the tests are given in the accompanying Table I.

The cup-drawing test, described in a paper presented at this meeting,[6] is an experimental method of evaluating drawability. The cup-drawing index gives the diameter of the largest blank which can be drawn into a cup and the percentage success in that size. The first number indicates the disk size in 32nds of an inch over the base diameter of 2 in. Larger indices indicate better test performance and presumably better drawing quality.

TABLE I.

Steel	Gage, in.	Carbon, per cent	Titanium, per cent	Ratio, Titanium to Carbon	Hardness		Cup-Drawing Index
					Vickers	Rockwell "B"	
No. 81.....	0.039	0.044	0.142	3.2	125	53	Less than 6–0%.
No. 82.....	0.045	0.044	0.142	3.2	139	60	Less than 6–0%.
No. 71.....	0.043	0.044	0.395	9.0	103	38	11–27%
No. 72.....	0.041	0.044	0.319	7.2	99.5	39	11–100% or 12–0%

The data in Table I indicate a wide difference in properties between the steels with high and low titanium-carbon ratios. Steels Nos. 71 and 72 have hardness values in the range normally encountered in deep-drawing sheets. Both steels have high cup-drawing indices and are above the average found for killed steels tested previously. Steel No. 72 gave less breakage in the test and probably has better drawability.

The data also show that the two steels with low titanium-carbon ratios had unusually high hardness values for sheet of drawing grade and very poor cup-drawing indices. No blanks of $2\frac{6}{32}$ in. diameter, the smallest tested, could be successfully formed into cups. This drawing performance is far inferior to that of average quality killed steel of deep-drawing grade.

MR. GEORGE F. COMSTOCK[7] (*author's closure, by letter*).—The discussion offered on this paper is much appreciated, and I should like to thank all those who participated in it.

Mr. McAdam's report of the occurrence of strain aging during the progress of high-temperature tension tests is worthy of careful attention by those engaged in such work.

The answer to Mr. Larson's question would seem to be "Yes," though the connection between the failure that he describes and low impact values is not very clear. This was apparently a case of severe internal stressing due to the thermal effects of welding, and loss of ductility through aging may well have been a contributing factor.

The excellent deep-drawing quality of the high-titanium steel sheets reported by Messrs. Boulger and Dahle is very interesting, and their discussion constitutes an extremely important addition to the paper. The sheets they tested were rolled, as they presumed, according to commercial mill practice, from full-sized ingots, and those with a titanium-carbon ratio of 7 to 9 were found to have no definite yield point and to be non-aging by the tension test method. Similar confirmation of the results reported in the paper has been obtained, since the paper was written, at two large steel mills with titanium steel rolled in sheet form under commercial conditions.

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[6] F. W. Boulger and F. B. Dahle, "A Test for Measuring Drawability of Deep-Drawing Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 503 (1943).

[7] Metallurgist, The Titanium Alloy Manufacturing Co., Niagara Falls, N. Y.

The detailed criticism of the paper offered by Messrs. Smith and Miller was especially appreciated, and their agreement with the paper in regard to the yield-point elongation being only an indication, and not a quantitative measure, of strain aging is noted. The grain sizes of many of the specimens tested are given in Tables III and IV, but it was not thought necessary to obtain this information for every steel used in the investigation.

The yield-point elongation was exceeded by the preliminary straining in all instances except for some of the tests where only 2 per cent strain was used. That this point of fundamental importance was not overlooked is indicated in the first column of page 528 of the paper. The yield strengths were of course determined during the first straining of all the specimens, and are reported for some of them in Tables VI to XI, inclusive. It should be appreciated that *all* the data obtained in this investigation could not be included in the paper. The opinion that a smaller number of steels should have been used is not shared by the author, as there are always unknown, as well as known, differences between individual heats of steel, and their effects can only be eliminated by checking a large number of different heats of each class of steel that is being studied. Instances in the current literature could be pointed out where incorrect conclusions have been drawn because of the testing of too few heats of a given class of steel.

It is admitted that "the conclusion that strain aging differs according as it is revealed by the return of a yield point or by the loss of impact strength is not in accord with current conceptions," but one object of the paper was to show that such conceptions need to be revised. Whether the difference is merely in degree or more fundamental in nature is not of great *practical* importance, and sufficient data to settle this question are probably not available. The important fact which the paper brings to light is that a given steel may be non-aging by one method of testing and aging by another; and therefore to call a steel merely "non-aging," as is often done, is meaningless unless the kind of aging, or the method of testing, is specified.

The criticism of the third paragraph of page 527 of the paper is not understood, since it is not inferred from only the elimination of the yield point in titanium steels that "such a steel is non-aging." This point is discussed in the first column of page 538. As indicated by the last sentence of Conclusion 1, there is no statement that a steel "classed as non-aging by the yield method may be classed as aging by the impact test," although steel No. 30 in Table III and Fig. 1 furnishes evidence of such a possibility.

No explanation can be offered for the different impact results in Table III and Fig. 1, except that straining by tension evidently had a different effect from straining by drawing through a die. The impact values given in Table III have been checked against the original data, and found to be correct. The low values found for steels Nos. 26 and 30 only in the not-aged condition are indeed surprising, and cannot be accounted for by the author; each result here is the average of two Izod tests. It may be noted that in both these steels the grain size was rather coarse.

It is possible that the failure of the impact tests to show aging in some instances might have been due to restricting the prior tensile straining to 5 per cent, but insufficient specimens were available to investigate the effects of other degrees of tensile strain. With the "current conceptions" as stated in the literature (see reference 12) indicating that only 2 per cent strain is required, it was decided that 5 per cent would be plenty, but according to the work-brittleness tests even that amount was not enough in all cases. Conclusion 2 is believed to cover this point adequately.

In reply to the final paragraph of Messrs. Smith and Miller, the fourteenth conclusion was reached because (1) deoxidation with aluminum, though more potent than titanium, was found not to eliminate the yield point; (2) decreasing nitrogen, like oxygen, was also found to be ineffective, steel No. 13 for instance showing a very definite yield point though having no soluble nitrogen; and (3) titanium added to the steel did not eliminate the yield point unless more than enough was present to isolate all the carbon as titanium carbide. The use of the words "*chief* cause," and "probably" in Conclusion 14 should be noted, indicating that the proof of this statement may not be absolutely flawless. A recent A. I. M. E. preprint by Low and Gensamer on "Aging and the Yield Point in Steel" provides interesting confirmation of that conclusion.

ON THE TRANSITION FROM A DUCTILE TO A BRITTLE TYPE OF FRACTURE IN SEVERAL LOW-ALLOY STEELS

By P. G. Jones[1]

Synopsis

This paper presents results of tests of several low-alloy ductile steels under conditions which tend to produce failure by brittle fracture. The embrittling conditions for a given hot-rolled ductile steel were considered to be primarily a high speed of straining, a nonuniform distribution of stress, a triaxial state of stress, and a low temperature. Other conditions, however, may contribute to the embrittlement of steel. Preliminary tests were made to determine what effect, if any, the direction of hot rolling and of overstraining had in causing a brittle fracture.

The data show that under some combinations of speed of straining, distribution and state of stress, and temperature the steel failed by brittle fracture. The suddenness of transition in the mode of failure from ductile to brittle fracture, however, varied considerably with the several steels tested. The influence of previous overstrain tended to increase the temperature at which the transition from a ductile to a brittle fracture occurred, other embrittling conditions remaining constant.

Factors Which Influence the Mode of Failure

The mode of fracture of a ductile steel member or specimen may consist of a fibrous, ductile type of fracture accompanied by plastic deformation (yielding) of a considerable portion of the specimen and by the absorption of a relatively large amount of energy. This type of fracture will occur in nearly all wrought (hot worked) steel in the usual standard tension test, in which the specimen is subjected to a uniform stress distribution, a uniaxial state of stress, a gradually applied load which causes a low rate of strain, and a normal (room) temperature.

On the other hand, almost any wrought steel may be made to exhibit a brittle type of fracture if it is subjected simultaneously to (1) a high rate of straining as in an impact test, (2) a nonuniform stress distribution as occurs in a notched specimen especially in resisting bending, (3) a triaxial state of stress as is likely to occur in a notched specimen, and (4) a low temperature. The influence of these factors on the mode of fracture has been studied by various investigators. An analysis of the results obtained by many early investigators including Maurer and Mailänder, Greaves and Jones, Moser and others is contained in a paper by McAdam and Clyne.[2]

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[1] Associate, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] D. J. McAdam, Jr., and R. W. Clyne, "The Theory of Impact Testing: Influence of Temperature, Velocity of Deformation, and Form and Size of Specimen on Work of Deformation," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 112 (1938).

A ductile type of fracture is assumed to occur when the resistance of the metal to yielding (often called the shearing yield strength) is less than its resistance to separation without accompanying yielding (often referred to as the technical cohesive strength[3]). On the other hand, a brittle fracture is assumed to occur when the technical cohesive strength is less than the shearing yield strength. Embrittling conditions, therefore, are assumed to be the conditions that will increase the shearing yield strength of the metal or lower the cohesive strength or both. A quantitative measure of the technical cohesive strength is practically impossible to determine and the influence of the factors that tend toward embrittlement is not known quantitatively, but qualitatively there appears to be justification for the assumption that increase in speed of straining and decrease in temperature promote embrittlement by increasing the shearing yield strength. Likewise a triaxial state of stress promotes embrittlement since it is accompanied by small shearing stress and shearing stress is associated with yielding. Other factors that may play a part in the embrittling action of the steel are residual stresses, internal discontinuities and cracks, grain size, grain orientation, and strain hardening as probably affected by previous overstrain, direction of rolling, etc.

Exploratory or preliminary tests are reported herein on the influence of the last two of the above conditions. Various combinations of the factors or conditions that tend toward embrittlement of the steel may be brought about in different ways, but only a small number of combinations has been used.

MATERIALS

The materials used for the tests included four low-alloy high strength steels A, B, C, and D, a structural steel E,[4] and a structural steel plate F.[4] The compositions of the steels are given in Table I. Properties for steels A, B, and E were previously reported by Collins and Dolan.[5] Steels A, B, and E in this paper correspond to steels A, B, and 36, respectively, in that report.

Except for the structural steel plate, the steels were received in the form of round hot-rolled bars. Steel D was $\frac{7}{8}$ in. in diameter and the others were $\frac{3}{4}$ in. in diameter. The steel plate was received in the form of a plate 18 in. wide, 1 in. thick, and 40 ft. long. A length of 16 ft. was placed in a large tension machine and overstrained 10 per cent in the direction of rolling. Transverse strips of the "as-rolled" steel 18 in. long and about 3 in. wide were cut from the long plate and overstrained in a direction perpendicular to the direction of rolling. Tests were made on specimens from the as-rolled steel and on specimens from the steel overstrained in each of the two directions. The test specimens used are

TABLE I.—COMPOSITION OF STEELS.

Steel	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent	Copper, per cent	Chromium, per cent	Nickel, per cent	Molybdenum, per cent
A	0.08	0.27	0.145	0.020	0.80	0.41	1.01	....	....
B	0.08	0.43	0.104	0.023	0.162	1.07	...	0.54	....
C	0.10	0.76	0.096	0.028	0.34	0.58	0.67	0.35	....
D	0.08	0.37	0.07	0.015	0.066	0.58	...	0.69	0.071
E	0.21	0.43	0.008	0.028	....	....	....	....	....
F[a]	0.20 to 0.35	....	....	....	....	....	....	....	....

[a] Heat analysis not available.

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[3] For a discussion of technical cohesive strength see, D. J. McAdam, Jr., "The Technical Cohesive Strength of Metals," *Journal of Applied Mechanics*, Am. Soc. Mechanical Engrs., Vol. 8, p. A-155 (1941).

[4] A.S.T.M. Standard Specifications for Steel for Bridges and Buildings (A 7 – 42), 1942 Book of A.S.T.M. Standards, Part I, p. 1.

[5] W. L. Collins and T. J. Dolan, "Physical Properties of Four Low-Alloy High-Strength Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 157 (1938).

shown in Fig. 1. The center section of the unnotched tension specimen was finished slightly undersize to insure the specimen breaking near the center. The notched tension specimens were finished in the lathe and the 60-deg. notch, having a 0.01 in. radius at the root, was formed with a special cutting tool. A special milling cutter was used to cut the 45-deg. notch on the notched bending specimens. A few tests were made in bending using the double width notched specimen as shown, but most bending tests were made with the single width (standard) specimen.

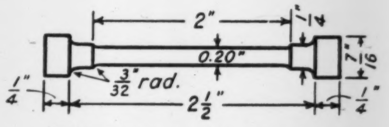
TEST METHODS

In the tests made, a Charpy impact machine having a capacity of 223 ft-lb. and a striking velocity of 17.4 ft. per sec. was used. For the bending specimens the distance between the anvils was 1.57 in. A special tension attachment on the Charpy machine was used for the tension impact specimens by means of which a close approach to axial loads was produced.

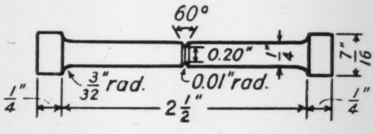
The low temperatures for the bending specimens were obtained by immersing the specimens in an acetone-dry ice bath for at least 5 min. at the desired temperature and then quickly transferring to the anvils of the testing machine. Temperatures above room temperatures were obtained from a bath of paraffin oil heated to the desired temperature. To reduce to a minimum the change in temperature of the tension specimen in removing the specimen from the cooling or the heating bath and placing it in the machine, it was found necessary to attach the tension rig and the specimen to the pendulum and then to cool or to heat the entire assembly, including a part of the pendulum. Although the time required for the tension tests was about 5 sec., the large mass of the assembly surrounding the specimen helped to minimize the change in temperature.

TEST RESULTS

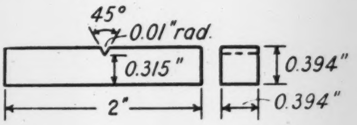
An indication of the embrittlement effect of any combination of embrittling conditions was obtained by plotting the energy absorbed by a given type of specimen in a Charpy impact test against the varying temperature and noting whether a rather sudden change in the energy absorbed took place. Little significance is to be attached to the numerical value of the energy absorbed as a measure of the ability of the steel to resist impact.



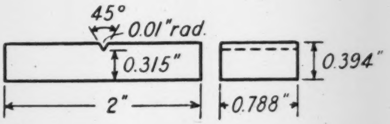
Tension Impact—Unnotched



Tension Impact—Notched



Bending Impact—Single Width



Bending Impact—Double Width

FIG. 1.—Details of Test Specimens.

Figure 2 shows that the three low-alloy steels A, B, and C and the plain carbon steel E exhibited a brittle fracture mode of failure when subjected to a rather high localized speed of straining (at the root of the notch) and to a nonuniform stress, a state of stress that probably was to some extent triaxial and a relatively low temperature of approximately 0 F., although the embrittling effect is noticeable at somewhat different temperatures for the different steels.

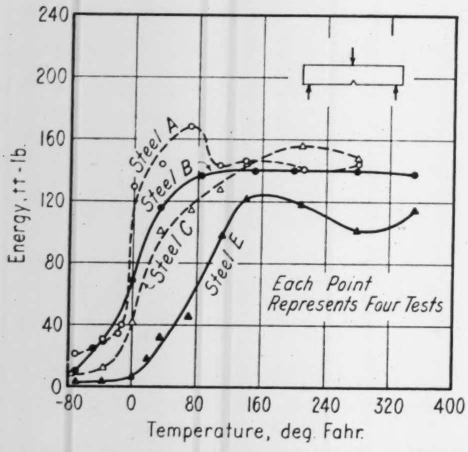


FIG. 2.—Results of Notched-Bar Impact Bending Tests on Steels A, B, C, and E.

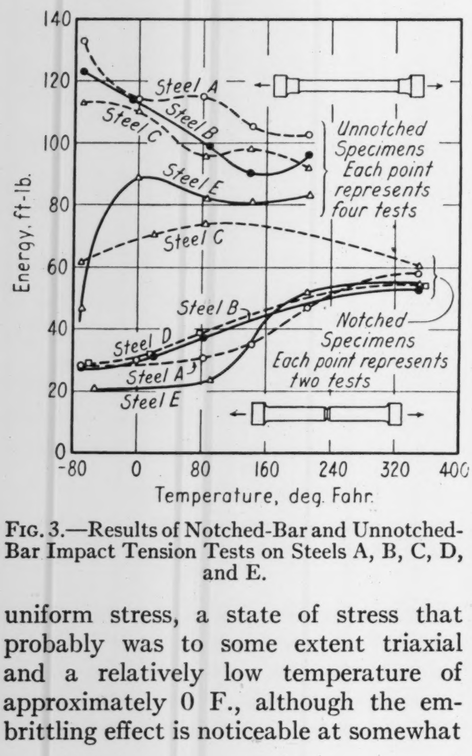


FIG. 3.—Results of Notched-Bar and Unnotched-Bar Impact Tension Tests on Steels A, B, C, D, and E.

On the other hand, the upper group of curves in Fig. 3 shows that these same three low-alloy steels are not embrittled at temperatures as low as −70 F. when subjected to uniform uniaxial stress and to a speed of straining which although high relative to static loading, is considerably less than the localized speed of straining in the notched bar bending specimens of Fig. 2. Steel E in Fig. 3, however, seems to be near to the combination of conditions that cause embrittlement.

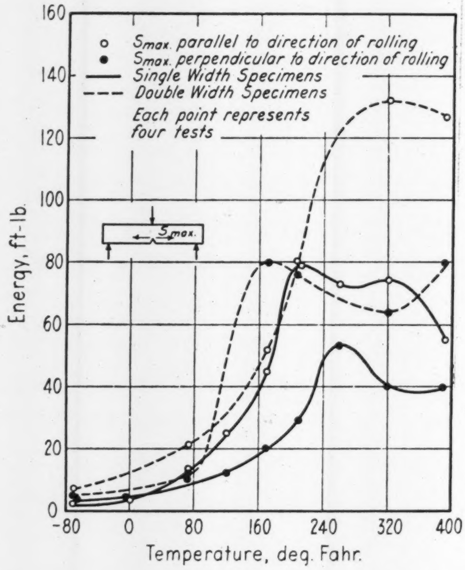


FIG. 4.—Results of Notched-Bar Impact Bending Tests on a Structural Steel Plate (Steel F) Using As-Rolled Steel.

The lower group of curves in Fig. 3 was obtained from the results of tests on notched tension impact specimens and are considered to represent conditions of embrittlement that are intermediate between those that prevailed in Fig. 2 and those that apply to the upper group of curves in Fig. 3. The speed of straining at the root of the notch was probably somewhat less than in the tests of notched bending specimens shown in Fig. 2. Although there was some evidence of embrittlement, the conditions under which this group of steels was tested were not sufficient to cause a rather abrupt change from a ductile to a brittle fracture.

In Fig. 4 steel F from the bridge steel plate was subjected to conditions that had been shown in Fig. 2 to embrittle the steels A, B, C, and E. Two other conditions were added, however, namely the direction of maximum stress was first parallel to and then perpendicular to the direction of rolling, and two widths of specimens were used. One set of specimens had twice the width of the standard specimens. The increase in width is considered to create a greater degree of triaxial state of stress in the central portion of the double-width specimen which is more favorable to the embrittlement of the steel. The main fact brought out by Fig. 4 is that the transition from ductile to brittle fracture occurred at much higher temperatures than did the steels shown in Fig. 2. No definite conclusions can be drawn from these data as to the embrittling influence of either direction of rolling or of double-width specimens. This problem needs further study.

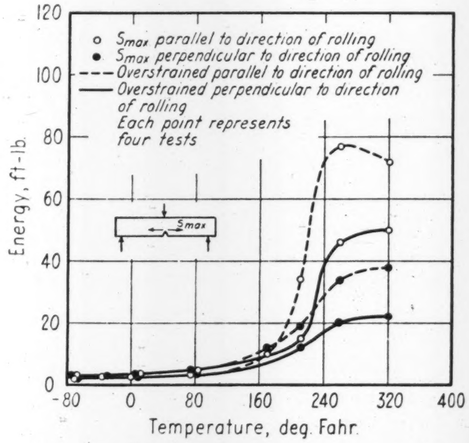


FIG. 5.—Results of Notched-Bar Impact Bending Tests on a Structural Steel Plate (Steel F) Using Overstrained Steel.

In Fig. 5 are shown curves representing the results of tests on notched impact bending specimens of the carbon steel plate (steel F) which had been overstrained 10 per cent in the direction of rolling and also on specimens which had been overstrained 10 per cent perpendicular to the direction of rolling. If one compares these curves to the curves in Fig. 4, which show results of tests on bending specimens of the as-rolled steel, it may be seen that overstraining increased the temperature at which transition from a ductile to a brittle fracture occurred.

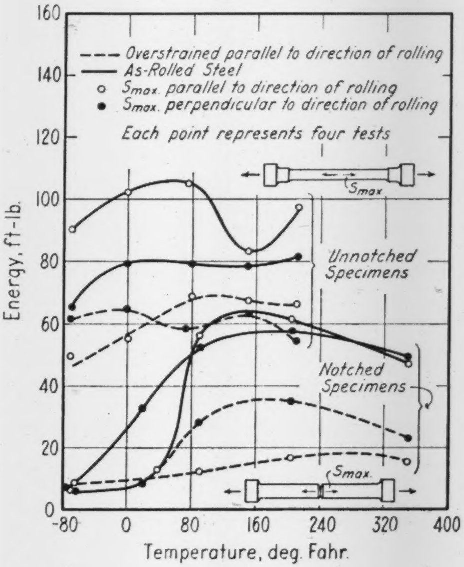


FIG. 6.—Results of Notched-Bar and Unnotched-Bar Impact Tension Tests on a Structural Steel Plate (Steel F) Using Both Overstrained and As-Rolled Steel.

The results of tests on notched and unnotched tension specimens of steel F are shown in Fig. 6. These tension tests were made on specimens of the as-rolled steel and on specimens of the steel overstrained 10 per cent in the direction of rolling. No transition temperature was determined for tests on unnotched tension specimens of either the overstrained steel or the as-rolled steel, although the upper group of temperature-energy curves in Fig. 6 indicate a downward trend for temperatures below 0 F. In the lower group of curves in Fig. 6, the tests on notched tension specimens of the as-rolled steel (solid lines) indicated that specimens tested so that the maximum stress was parallel to the direction of rolling would show a rather sudden transition from a ductile to a brittle fracture at a higher temperature than specimens tested so that the maximum stress was perpendicular to the direction of rolling. In this same group of curves the tests on notched tension specimens of the overstrained steel (broken lines) indicated rather brittle fractures throughout the range of temperatures used. This was especially true for those tests on specimens in which the maximum stress was parallel to the direction of rolling. Referring to Fig. 5, one may see that the curve representing results of tests on bending specimens cut from this same overstrained steel and also tested so that the maximum stress in bending was parallel to the direction of rolling (upper curve) indicates a rather sudden transition from a rather ductile fracture to a very brittle fracture.

The curves shown in Figs. 2 to 6 are drawn through points which represent the average of four duplicate tests except for the notched tension specimens of steels A, B, C, D, and E as shown in Fig. 3, in which each point represents the average of two tests. In Fig. 7 are shown curves representing the results of tests on notched impact bending specimens of steels A and E in which each point represents a single test. (These curves were previously shown in Fig. 2.) From these data one may observe the "scatter" of the test results at various temperatures for steel A which exhibits a rather sudden transition from a ductile to a brittle fracture, and for steel E which shows a more gradual transition. It should be noted that the curves in Figs. 2 and 7 are plotted to a different scale than those in the other figures.

Smooth curves were drawn through the points that represent average values in order to take advantage of trends which would be less evident if straight lines were drawn between the points. Although some specimens tested in impact bending at temperatures above the critical temperatures did not break into two pieces, the results of these tests are included in this report because they do indicate the ductile type of failure which was exhibited. However, little significance should be attached to the actual numerical value of energy absorbed, even for comparative purposes.

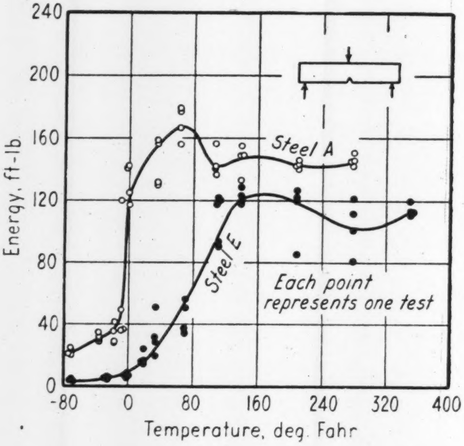


FIG. 7.—Results of Notched-Bar Impact Bending Tests on Steels A and E.

In summarizing the results of this preliminary investigation, it may be said that under some combinations of speed of straining, distribution and state of stress, and temperature the ductile low-alloy steels failed by brittle fracture. The suddenness of transition in the mode of failure from ductile to brittle fracture, however, varied considerably with the several steels tested. Sufficient knowledge is not available to predict the various combinations of factors or conditions that will cause a ductile steel to fail by brittle fracture. Much more experimental work needs to be done in this field. The influence of previous overstrain of structural carbon steel tended to increase the temperature at which the transition from a ductile to a brittle fracture occurred. No definite conclusions can be drawn from the data concerning the embrittling influence of the direction of hot-rolling of the structural carbon steel.

This paper should be considered in the nature of a preliminary or progress report. Work on the investigation was stopped before the project was completed, because of war conditions. However, service failures involving brittle fracture of so-called ductile metals seem to justify a partial report on the work planned.

Acknowledgments:

The author wishes to acknowledge the help of D. C. Koehler, R. F. Skelton, and F. Seyfarth, students in the College of Engineering who carried out various parts of the investigation, mainly as theses under the supervision of the author. Acknowledgment is also made to F. B. Seely, Head of the Department of Theoretical and Applied Mechanics, for his help and suggestions in planning the tests and in preparing this paper.

DISCUSSION

Mr. J. J. Kanter.[1]—There is one point in connection with some of the data that the author has shown that does not seem to check with the general experience in testing metals for brittle breaks through the range of transition from ductile type to brittle type of fracture. In this range, scatter of results is usually observed and the smooth curves shown in Fig. 2 are at least not characteristic.

Recently we did some careful testing in an effort to throw some light on this very point. Ten specimens were tested at each of a number of established temperatures covering the transition range; at each of those temperatures it was found that both ductile and brittle type failures occurred. The principal feature that characterizes this variation is that as the temperature lowers the greater is the proportion of brittle failures to ductile failures. There seems to be an almost direct proportionality between lowering temperature and the probability of a brittle failure.

It would be of value if the author made clear whether his points represent single or average values,—and if average value what character of scatter in the transition was observed.

Mr. S. L. Hoyt.[2]—I have been very much interested in this paper. There are two points in particular that I should like to comment on.

One was the significance which appears to be given to the speed of deforming. As far as I know, except for a few steels this is not particularly critical until bullet velocities are reached. In the summary of the paper the speed of deformation is given the same weight as the other variables, which seems to me a grave error.

As to the theory involved here, the author pays no attention to the Ludwik hypothesis, which is the one based on the assumption of a cohesive strength and its relation to the shearing strength of the material. I should like to see the author consider his results in the light of the Ludwik hypothesis which is a very satisfactory hypothesis to use here.[3]

The work itself seems to be pretty much of a substantiation of previous work which shows that notched bars are brittle at low temperatures, whereas ductile behavior is experienced at room temperature.

Mr. P. G. Jones[4] (*author's closure, by letter*).—The author agrees with Mr. Hoyt that it may be a grave error to give speed of deformation the same weight as the other variables affecting the mode of failure at velocities of deformation lower than bullet velocities. In the summary of the paper the relative importance of the variables affecting the mode of failure is not given and the speed of deformation is not intended necessarily to be given the same weight as the other variables. However, it is a factor which should be considered even if its effect is not particularly critical until bullet velocities are reached. In a recent paper by Hollomon and Zener[5] there are presented considerable experimental data on the effect of speed of deformation at relatively low speeds. Although these data show that the effect of speed of deformation is not critical in the sense that a sudden transition in type of failure is observed, they do show that the importance of this factor cannot be ignored. It must be remembered that although the apparent velocity of deformation obtained in many instances of high-speed loading is considerably less than bullet velocities, the rate of deformation (specific velocity) at a point of stress concentration in a notched member may be very high and may approach bullet velocities.

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[1] Materials Research Engineer, Research and Development Laboratories, Crane Co., Chicago, Ill.

[2] Technical Adviser, Battelle Memorial Institute, Columbus, Ohio.

[3] S. L. Hoyt, "Notched-Bar Testing and Impact Testing," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 143 (1938).

[4] Associate, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

Although no reference is made to the Ludwik hypothesis as such, the conception of a cohesive strength and its relation to the shearing yield strength of the metal is discussed in the paper. The results of the tests do not verify the Ludwik hypothesis but they are not inconsistent with it. The rather gradual transition from a ductile to a brittle type of failure which was observed in some of the tests can be explained if consideration is given to the effect that plastic deformation may have on the value of cohesive strength and the resistance to deformation. In these tests the cohesive strength of the metal was sufficiently greater than the shearing yield strength to support the normal stress up to the time that plastic action started. The combinations of conditions imposed in the various tests were such that the plastic deformation caused a relative change in cohesive strength and resistance to deformation, but this change was different for different temperatures and consequently there was some plastic action in all tests but the degree varied with temperature.

As pointed out by Mr. Kanter in his discussion there are some combinations of conditions which may produce either a ductile or a brittle type of failure, but it is questionable that such results are characteristic although they certainly are more spectacular. The conditions of instability which produce these results are evidently such that the value of the cohesive strength is so low that the normal stress produces brittle cleavage at the same time that the shear component of the applied stress overcomes the resistance to deformation. Also under these conditions of instability the ratio of cohesive strength to resistance to deformation is not affected by plastic deformation so that when deformation starts it is able to continue and result in a ductile type of failure.

Mr. Kanter has evidently overlooked the notation which appears in each graph giving the number of tests represented by each point. Since each point represents the average of a number of tests, Fig. 7 was included to show the character of scatter in the transition range.

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[5] J. H. Hollomon and C. Zener, "High Speed Testing of Mild Steel," *Preprint No. 13*, Am. Soc. Metals, (1943).

CHROMIUM PLATE IN ENGINEERING APPLICATIONS—ITS THICKNESS AND FINISHING

By T. G. Coyle[1]

Synopsis

Thickness values and finishing treatments for a number of important engineering uses of chromium plate are recommended and discussed. The information given complements the operating recommendations on plating methods outlined in the recently accepted A.S.T.M. Tentative Recommended Practice for Chromium Plating on Steel for Engineering Use (B 177 - 43 T).[2] Plate thickness values generally found best for the more commonly used applications are included; and discussed specifically are practices to be recommended for many wear resistance requirements on parts such as tools, gages, molds, drawing dies, and plugs. Suggestions and recommendations covering preferred plating conditions for certain cases, and finishing treatments, such as grinding and lapping are also included. Some discussion is devoted to the function of the chromium plate in different applications, by way of generalizing for guidance in other applications not discussed in detail. Views on the practical limitations of setting up complete specifications for this general class of chromium plating are stated.

The recently accepted A.S.T.M. Tentative Recommended Practice for Chromium Plating on Steel for Engineering Use (B 177 - 43 T)[2] outlines quite completely methods and operating conditions for electrodepositing chromium directly on steel. The details of the procedure and operating techniques there described may be considered to apply to chromium plating on steel generally, without special reference to the type of article to be plated or to the particular function the chromium plate is to perform. It should not be concluded from this, however, that all steel articles are chromium plated alike; on the contrary, for best results different applications require different thicknesses of deposit, and some require special plating conditions and finishing treatments.

The purpose of this paper is to give information on such requirements, and to treat more specifically some of the commonly used applications. In short, the information given here on "how much plate" is intended to complement the recommendations on "how to plate" as outlined in Tentative Recommended Practice B 117.

Tests and Data

While a great amount of testing in both field and laboratory has been carried out on the various applications of chromium plate on steel, no attempt is made here to describe these tests or to include test data. The recommendations on plate thickness and plating conditions or plate treatment are based principally on experience in the field over many years and represent the most commonly accepted practices.

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[1] Technical Director, United Chromium, Inc., New York, N. Y.

[2] 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 327.

LIMITATIONS

As will be apparent from the following, setting up complete specifications on chromium-plate applications for engineering use is hardly practicable. While a general procedure for the plating process proper can be outlined, as shown in Tentative Recommended Practice B 177, the thickness and finish requirements vary not only from one class of service to another, and from one class of articles to another, but even among different articles in the same type of service. Conditions of service, such as load, abrasion, temperature, and corrosion, not uncommonly differ for the same class of article and require diverse amounts and treatments of plate. Pump rods, for example, working in different type wells, some under severe corrosive conditions, others under severe abrasive conditions, might require different handling for the different service conditions; or drawing dies and plugs on different drawing operations might require different thicknesses of deposit for best results in each case. Conversely, the condition of the article itself as to size, shape, or construction might dictate the kind of finish or the amount of plate to be used. An example here is a cylinder, or a gage, worn oversize or out of round, thereby requiring a heavier plate or a different method of processing than would otherwise be recommended.

Each application is, in varying degree, an individual problem. The information in the following on plate thickness values and plating conditions or finishing treatments should be considered in this light, and taken as a general guide, with any individual case possibly requiring a departure from the recommendations given.

WEAR-RESISTANCE APPLICATIONS

Most applications of chromium plate on steel for engineering use are made primarily to obtain better abrasion resistance and to decrease wear on the plated article or part. Into this category fall most tools, cutters, gages, dies, and machine parts, as well as bearing surfaces, printing surfaces, rods, plungers, and similar equipment. In the latter group the corrosion resistance of the chromium also contributes in important degree to the improved performance of the plated part, but the principal function of the plate is to withstand abrasion. Several of these wear resistance applications of chromium plate are discussed below.

Gages:

Plug gages, ring gages, and other gages of various types are generally finished with a chromium plate 0.001 to 0.0015 in. thick (0.002 to 0.003 in. on the diameter). Beyond giving a deposit of sufficient thickness to withstand the load and pressure of the gaging operation, the object is to have a deposit of thickness greater than the wear tolerance of the gage. In this way a chromium surface is maintained throughout the accuracy life of the gage, and at the same time the underlying steel basis metal is protected from wear or scoring or other damage so that the gage can be resized very easily by dissolving ("stripping") the remaining chromium and replating the gage. This practice, it should be noted, applies to many of the other applications discussed below, in particular drawing dies and mandrels, many machine parts, and certain tools.

In some gaging operations, where tolerances are extremely small or the gage must have a sharp working edge, the chromium plate thickness should be limited to much less than the values given above. The thickness should be only of the order of 0.0001 to 0.0003 in. (0.0002 to 0.0006 in. on the diameter). The purpose here is to avoid chipping or spalling of the plate that might occur on the sharp edge if heavier deposits were used. The thinner deposits, even though they may afford lesser increase in number of gaging operations than the heavier plate, still give the advantages of the chromium working surface, and are desired for this reason as well as for protection of the steel and the resizing practice the plating makes possible.

In practice, the usual procedure is to grind the gage undersize by the amount of plate thickness required, plate oversize by an amount sufficient to give grinding stock, and finally grind or lap back to gage size, leaving a finished chromium deposit of the required thickness. In cases where the thin (0.0001 to 0.0003 in.) deposits are used, the gage may be plated directly to size with sufficient accuracy, and in some cases also in the greater thickness; but generally it is more practical and economical to get the accurate sizing required by grinding or lapping to size after plating. On gages, a plate 0.002 to 0.004 in. oversize in diameter usually provides sufficient stock for grinding, allowing for eccentricity in successive grinding set-ups, but the amount actually required may be more or may be less depending on the size of the gage as well as other factors, such as grinding equipment and methods.

The finish on the gage should be smooth and lustrous. As a rule, the ground, lapped, or honed finish is adequate, but a light buffing operation for the final finish may be desirable.

A stress-relief heat treatment after plating is advisable, especially for the gages with sharp corners and edges and gages having higher hardness values before plating. See Recommended Practice B 177 for recommendations as to the hardness of steel and stress-relief heat treatment after plating.

Tube-Drawing Dies and Plugs:

As in the case of gages, many tube-drawing plugs and dies are ground undersize, plated oversize, and ground to size for use. However, unlike gages, dies often are used without grinding or finishing after plating, because the size tolerances are usually greater and the first few passes of the work over the die produces a satisfactory finish. Dies and plugs are given stress-relief heat-treatment after plating.

The best thickness of chromium for drawing plugs and dies depends on the material to be handled, the speed of drawing, and the reduction in each draw. A typical thickness of deposits is 0.003 in. (0.006 in. in diameter). In some cases of large dies or large tolerances thicknesses as great as 0.008 to 0.010 in. are applied, and in others deposits as thin as 0.0005 in. are used. These lower thicknesses are applicable to some of the smaller plugs and dies, and have been used to avoid the extra grinding operations often necessary with the heavier deposits.

Forming and Stamping Dies:

Like in tube drawing, the thickness of plate on forming dies depends largely on the material being worked, and the particular operation, but generally the plate thickness is less than that used for tube drawing. For simple forming operations, a thickness of 0.001 to 0.002 in. is generally satisfactory. For deep drawing and working thin-wall sections, thinner deposits are often better, down to 0.0002 to 0.001 in. The same applies to stamping, shearing, and blanking operations; similarly, coining dies should have the thinner deposits, 0.0002 to 0.0005 in. A generalization for these types of dies and tools is that for those operations involving sharp impact or cutting action, the thickness of deposit should be kept at a minimum consistent with getting economical and efficient performance from the plating operation. Wherever there are sharp edges or impact involved, the heavier deposits show greater tendency to spalling or chipping.

Molding Dies:

Except where highly abrasive materials are being molded, the plate thickness on molding dies need not be very great. For most plastics, thicknesses of 0.0002 to 0.0005 in. are generally satisfactory; for clays and similar materials, heavier deposits up to 0.002 in. thick are recommended; and on large work, still higher values.

A very important factor in good results from plated molds is the finish of the molding surface before and after plating. Except where dull or special finishes are required on the molded product, the mold should be finished to a high luster by lapping, polishing, or buffing.

Cutting Tools:

The statement in the Section on Forming and Stamping Dies about plate thickness in cutting operations should be repeated here: namely, in such operations the thickness of deposit should be kept at a minimum consistent with getting most economical or efficient performance from the plating operation. Cutting surfaces in general should have relatively thin deposits to minimize tendency to spalling or chipping; usually not more than a few ten-thousandths inch thickness, and in many instances less than 0.0001 in., as little as 0.00005 in.

With cutting tools especially, the best thickness of plate depends not only on the operating conditions, such as speed and amount of cut, but also very largely on the material being cut. The best thickness for cutting steel might be quite different from the best thickness for cutting hard rubber or plastics or even metals like aluminum, zinc, brass, or copper. A generalization here is that the harder the metal being cut the thinner should be the chromium deposit. The thinner deposit sacrifices the longer wear a heavier plate might give, but in cutting on harder and tougher materials, the heavier deposits show greater tendency to fail by chipping or spalling. A suggested range of plate thickness for cutting tools in general is 0.00005 to 0.0001 in. for hard steels, 0.0001 to 0.00025 in. for mild steels, cast iron and non-ferrous metals like aluminum, brass, bronze, copper, and zinc; and 0.00015 to 0.0005 in. for nonmetallics such as rubber, slate, plastics, wood, and fiber.

Inasmuch as most cutting tools have thin cross-sections and are made of high speed or high-carbon steels, heat treatment for stress relief after plating is especially recommended. Attention should be given also to the hardness of the tool before plating, to see that it is within recommended hardness limits (C62 to C64).

Another precaution in handling cutting tools is to dress the cutting edge carefully before plating. It should be made free from any slight burring or feathering; this may be accomplished by a slight honing operation, by hand, using a fine stone.

A few of the more commonly plated cutting tools deserving special comment are described in the following.

Twist drills are chromium plated over their full length. The flutes should be completely covered with chromium. With the plate so thin (0.00005 to 0.0005 in. as discussed above) generally the drill needs no special sizing. Before being put into use the drill should be dressed to remove chromium from the point and to provide proper clearance on the back faces. The drill is sharpened as required, and one plating should last the life of the drill.

Reamers are usually plated all over and then ground to proper size and clearance. The grinding leaves chromium on only the face of each blade. The plate thickness on reamers may run a little heavier than on other cutting tools, from about 0.0001 to 0.0005 in.

Files are given thin deposits and plated only enough to give complete coverage. The current required depends to some extent on the condition of the file; a current density of 4 to 6 amp. per square in. of superficial area is recommended for about a 5 min. plating time at 100 to 120 F. Heavy deposits dull the file and should be avoided. The files are ready for use as plated except for the stress relief heat treatment that should follow the plating.

Broaches, *milling cutters*, *gear cutters*, *knives*, *threading dies*, *taps*, and *thread chasers* are all examples of cutting tools commonly chromium plated. The general comments and suggestions at the beginning of this section apply to these tools.

Burnishing Bars:

Burnishing bars should be given a deposit of 0.002 to 0.003 in., although deposits as light as 0.0005 in. are sometimes used. The plate should be finished smooth by grinding, lapping, or buffing, and the bar should be given a stress-relief heat treatment.

Pump Shafts:

For most pump shafts, a chromium plate thickness of 0.0005 to 0.0015 in. (0.001 to 0.003 in. on the diameter) is satisfactory; but for use under high-pressure packing, heavier deposits should be applied, about 0.003 in. thick (0.006 in. on the diameter). On large shafts, the work may be plated oversize (about 0.005 in. thickness) and ground back to size. The finish in all cases should be smooth, preferably buffed to a high luster.

Pump Plungers, Rods, and Rams:

Pump plungers and hydraulic rams are handled in much the same way as pump shafts. The plate thicknesses may run higher, 0.005 to 0.010 in., and in extreme cases as high as 0.100 in. All bearing surfaces of this type running against packing should be finished very smooth before use.

Hydraulic Equipment:

There are many hydraulic parts chromium plated, most of them similar to pump shafts or rams in plate requirements. Where they run through packing and a deposit of 0.0005 to 0.00015 in. is satisfactory, they may be plated to approximate size and buffed. Where they function inside a cylinder with close tolerance, they should be plated to oversize and ground to size, with a residual plate of 0.001 to 0.003 in. in thickness (0.002 to 0.006 in. on the diameter).

Machine Parts:

The many machine parts regularly chromium plated include cams, feed screws, spindles, lathe centers, crossheads, guides, and similar pieces. For the most part, these take approximately 0.0005 to 0.003 in. in thickness, finished smooth to these dimensions by grinding or lapping to size after plating.

Printing Plates and Rolls:

Steel engraved printing surfaces require a deposit of only 0.0002 to 0.0005 in. for good service. Heavier deposits should generally be avoided to prevent loss of detail in finely etched areas.

CORROSION-RESISTANCE APPLICATIONS

In a number of the wear-resistance applications outlined above corrosion resistance also is an important factor. Protection of the molding die surfaces against pitting and erosion is very important; and on pump shafts, rods, and plungers, resistance of the chromium to corrosion from the packing or material handled contributes in an important way to the successful performance of the plated member. There are many applications where the opposite condition obtains: where the part is chromium plated primarily for corrosion resistance, and wear resistance though possibly important is a lesser consideration in using the chromium plate.

The plate thickness used for corrosion resistance applications varies over wide limits, from 0.0002 to 0.0005 in. for bearing rollers operating in oil, to 0.010 to 0.015 in. or more on large rolls and operating equipment. The applications for corrosion resistance are numerous and varied and include press plates, calendar rolls, table rolls, graining rolls, coating rolls, lehr rolls, drying rolls and drums, chemical handling and mixing equipment, tanks, food processing equipment, oil refinery equipment, and numerous other pieces of industrial equipment, besides many engine and machine parts.

SALVAGE APPLICATIONS

The number of salvage applications is practically unlimited. Salvage plating includes articles of all sizes and shapes, representing parts worn out in service as well as parts machined off-size in fabrication. Obviously, the thickness requirements in such applications are determined by the amount the part is off-size, except where the maximum plate thickness that will function satisfactorily is less than the amount the undersize part has to be built up.

CONCLUSION

The information on deposit thicknesses and plate treatments as given in the foregoing illustrates the statements made earlier regarding limitations to specifications on chromium plating for engineering use. Hard and fast recommendations on plate thickness can hardly be made for a single type application, let alone a specification to cover the many and diverse applications of chromium plate in its engineering uses. It is possible, however, to give information of a general nature for the various uses, to serve as a guide to final determination, by trial, of the best practice for individual cases. It is believed that the above recommendations and suggestion may serve this purpose.

PROPERTIES OF PLATED LEAD COATINGS ON STEEL

By K. Gustaf Soderberg[1]

Synopsis

This paper has been prepared to assist users of the new A.S.T.M. Emergency Specifications for Electrodeposited Coatings of Lead on Steel (ES-31)[2] in choosing the proper type of coating for each individual application.

The paper presents information gleaned from the literature together with private findings not published heretofore.

Since exposure test data for lead coatings are somewhat meager, there are included some test results on the corrosion of solid lead and on the corrosion of steel and other metals in contact with lead. These results throw further light on the general subject.

Outdoor Atmospheric Exposure

The most complete outdoor tests of lead coatings on steel were made by Clarke[3] in the industrial atmosphere of Woolwich, England. The lead was deposited in a perchlorate bath on panels of sheet steel. Nothing is said about how the panels were held during exposure, but one suspects a vertical position which is commonly used in England. The results are given in Table I.

This report is interesting for several reasons. While porosity existed in the 0.001-in. thick coating, the pores closed up or became filled and ineffective on continued exposure. Such apparent disappearance of pores did not take place in the thinner coatings. Thickness figures which fall in the neighborhood of 0.001 in. appear again in other references.

The present author has a number of lead-plated panels exposed at 45-deg. facing south on a roof 200 to 300 ft. from a railroad in Detroit, Mich. The plating was done in a lead fluoborate bath with glue as addition agent. Some rust appeared on panels with 0.00025 in. lead after 3 months, and after 6 months an average of 6 per cent of the top surfaces and 45 per cent of the bottom

TABLE I.—OUTDOOR CORROSION OF LEAD-PLATED STEEL AT WOOLWICH, ENGLAND.

Thickness of Lead, in., avg.	Exposed 6 months	Period of Exposure 1 yr.	3 yr.
0.0001....	Rusted all over	....	....
0.00025..	One third of surface rusted	Rusted nearly all over	Flaky rust all over
0.0005...	Few very small rust spots	Small rust patches	Rust all over, smooth, adherent
0.001....	Few very small rust spots	Few very small rust spots	No apparent rust, good conditions[a]

[a] Further comments—"The coating had become blackened and a slight degree of roughness had developed due to microscopic nodules of corrosion product. No rust was apparent, except at a scratch originally made through the coating; the rust was hard, dark in color and very localized."

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[1] Technical Director, The Udylite Corp., Detroit, Mich.

[2] Issued May 29, 1943, published in separate pamphlet form.

[3] Clarke, *Journal*, Electrodepositors' Technical Soc., p. 141 (1939).

surfaces were rusted. After 10½ months the percentage rust was 7 and 73, respectively. Both top and bottom of the panels with 0.0005 and 0.001-in. lead were substantially free from rust after 10½ months.

For comparison it may be noted that a 0.0002-in. thick zinc coating prevented rust for about 7 to 8 months on another roof close to the one mentioned above.

Two processes take place on the surface of a lead-plated steel article when it is exposed to the weather: one is the corrosion of the lead coating itself, the other the corrosion of the steel through pores in the coating. It is evident that as the coating itself is weathered away, more pores are opened up to the steel.

TABLE II.—OUTDOOR CORROSION OF SOLID LEAD IN THE UNITED STATES.

Test Site Type of Atmosphere	Years of Exposure	Thickness Corroded, in. × 10^{-6}			
		Lead U	Nickel T	Copper A	Zinc HH
LaJolla, Calif. (foggy seacoast)	9.14	162	44	521	480
Key West, Fla. (seacoast)	8.78	217	47	199	151
Altoona, Pa. (industrial)	9.25	267	1520	464	1750
New York, N. Y. (industrial near seacoast)	9.38	167	1290	471	1897
State College, Pa. (rural)	9.27	189	62	228	358
Phoenix, Ariz. (desert)	3.85	95	12	54	46

One may expect that the rate of weathering of the electroplated lead would be of the same order of magnitude as that of the solid lead. Data on the latter are available from several sources: the work of the Society's Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys presented in this year's report[4] and also tests conducted by Hudson[5] and Friend[6] in England. Committee B-3 exposed vertically 9 by 12 by 0.035-in. weighed panels in a number of test locations. After nearly ten years, the corrosion products were removed and the panels were reweighed. Hudson exposed 2½ by 2 by 0.04-in. panels for 1 yr. only and followed the same general procedure. Friend exposed, near one of Hudson's stations, weighed rods about 24 in. long and 1⅛ in. in diameter, with their axis in horizontal position. After 7 yr., the bars were cleaned and reweighed. The weight loss per unit area, all surfaces included, have been recalculated into average decrease in thickness, and are given here in Tables II and III.

TABLE III.—OUTDOOR CORROSION OF SOLID LEAD IN ENGLAND.

Type of Atmosphere	Exposure Site	Years	Thickness Corroded, in. ×10^{-6}			
			Lead	Nickel	Copper	Zinc
Rural	Cardington	1	56	45	76	117
Suburban	Bournville	1	77	96	115	197
Urban	Birmingham	1	145	230	158	376
Urban	Birmingham	7	238	1323	644	1512
Industrial	Wakefield	1	74	218	156	261
Marine	Southport	1	70	111	148	199

To identify the atmospheres further Hudson collected some data on the dust which settled in the different locations during the time of the tests. These data are given in Table IV.

It is evident that the atmosphere in all the English exposure sites were rather heavily contaminated. The amount of sulfur trioxide appears large enough that the corrosion products formed on the lead were probably always sulfates.

The excellent performance of the lead as compared to that of the zinc is notable in all locations except two of the American sites, Key West and Phoenix, both of which attack the zinc very slowly. Where corrosion is at all severe, the attack on the lead is definitely less than

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[4] See p. 135.

[5] Hudson, *Transactions*, Faraday Soc., Vol. 25, p. 235 (1929).

[6] Friend, *Journal*, Inst. Metals, Vol. 42, p. 149 (1929).

TABLE IV.—AMOUNT AND ANALYSIS OF DUST AT EXPOSURE SITES.

Site	Monthly Deposit, g. per 100 sq. m.				
	Insolubles	Solubles	SO_3	Cl	NH_3 in Solubles
Cardington	141	274	51	17	2
Bournville	244	307	62	29	1
Birmingham	717	497	163	67	11
Wakefield	222	571	198	93	7
Southport	203	325	66	103	1

TABLE V.—PROTECTION OF IRON OUTDOORS BY COUPLE ACTION OF OTHER METALS OF EQUAL AREA.

Test Site	Couple			
	Pb/Fe	Ni/Fe	Cu/Fe	Zn/Fe
LaJolla, Calif.	2.47/0.52	......	0.25/0.05	2.18/0.08
(foggy seacoast)	+		+?	+
Sandy Hook, N. J.	1.78/0.45	1.86/0.77	1.92/0.95	2.60/1.62
(smoky seacoast)	+	+	+	+?
Key West, Fla.	1.44/0.36	8/1.10	1.00/1.86	2.00/0.02
(seacoast)	+	+?	−	+
Pittsburgh, Pa.	1.39/1.10	1.24/1.04	0.63/1.74	1.77/0.02
(industrial)	+?	+?	−	+
Altoona, Pa.	1.42/1.01	0.84/0.68	0.43/1.38	1.26/0.45
(industrial)	+	+??	−	+
Rochester, N. Y.	1.43/1.38	1.69/1.13	0.48/1.46	0.30/0.04
(industrial)	+??	+?	−	+?
New York, N. Y.	1.06/0.98	0.80/0.71	0.62/1.44	1.79/0.03
(industrial, near seacoast)	+	+??	−	+
State College, Pa.	1.56/0.62	0.50/1.06	1.19/1.36	3.00/0.31
(rural)	+	−	−??	+
Phoenix, Ariz.	1.24/0.67	0.50/1.07	1.60/1.12	4.10/0.07
(desert)	+	−	+?	+

NOTE.—The first figure of the ratio represents the weight loss of the metal when coupled with iron divided by the weight loss of the metal when coupled with itself. The second figure represents the weight loss of iron when coupled with the metal in question divided by the weight loss of the iron when coupled with itself. Where the first figure is larger than 1.00 and the second figure is smaller than 1.00, the metal in question is anodic to the iron and tends to protect it and the couple has been marked +. Where the reverse is true the metal accelerates the corrosion of the iron and the couple is marked −. In some instances both figures are larger or both are smaller than 1.00. If in this case the first figure is substantially larger than the second the couple has been marked +? and if the second figure is substantially larger than the first figure the couple has been marked −?. Where the differences are small two question marks are used.

that on copper. Lead corrodes more slowly than nickel only in atmospheres high in sulfur trioxide. Of particular interest is the comparison between the 1 and 7-yr. exposures in Birmingham. Although Hudson comments that Friend's exposure was more shielded than his own, it appears probable that the lead sulfate, etc., film formed has considerable protective value in this atmosphere.

Some evidence relative to the effect of porosity of lead coatings may be obtained from the galvanic couple tests of A.S.T.M. Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys.[7] In these tests, washers of different metals were bolted together in such a way that the only metallic contact was between the washers being tested and that the only surfaces subjected to the atmosphere were those of the edges of the washers. This provides a couple of two metals of equal area. Unfortunately, the couples deteriorated during the tests in a number of different outdoor locations and the exposed areas did not remain constant. For this reason it is most difficult to draw very definite conclusions. The results are given in Table V.

One may conclude that lead coatings have greater tendency to protect the underlying iron electrochemically through pores in the coating in uncontaminated or seacoast atmospheres than in an industrial atmosphere. The presence of chlorides in industrial atmospheres seems to increase the protective ability of the lead (for example in Sandy Hook, N.J. and New York City). In a strictly industrial atmosphere lead does not offer much, if any, electrochemical protection and may actually cause acceleration of the corrosion of the steel.

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[7] Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 247 (1939).

Nickel, copper, and zinc were included for the sake of comparison. It is evident from the table, and still more so from the original data, that while lead is more anodic than either nickel or copper, it is not by any means comparable with zinc in this respect.

It may be noted here that the porosity of the coating depends on the base metal and the structure of the lead deposit. Light burnishing and even scratch brushing assist in making thin lead coatings less porous.[8]

Kurrein[9] states that while coatings as thin as 0.00012 in. have proved pore-free in ferricyanide tests, a thickness of 0.00035 in. may be considered as providing lasting rust protection.[10] Blum, *et al*,[11] on the other hand speak of 0.003 to 0.005-in. thickness of lead from well-controlled lead fluoborate baths as being necessary for complete freedom from porosity.

INDOOR ATMOSPHERIC EXPOSURE

With respect to indoor exposure we are again indebted to Clarke.[3] He exposed a number of lead-plated steel panels during the winter months in a small unheated building in Woolwich, England. The window was left partly open to the polluted and dusty outside air. During nights the samples were moved into a closed vessel containing a layer of water. With the overnight drop in temperature this ensured a damp condition being reached on the metal surfaces.

The appearance after 6 months of the lead-coated samples (from perchlorate bath) and, for comparison's sake, of nickel, copper, and zinc-coated specimen are given in Table VI.

Unfortunately, no strictly comparative tests with solid lead are available. However, Vernon[12] reports some indoor atmospheric tests in a basement in South Kensington, England. The basement was heated with steam radiators during the winter months while these tests were going on. The dew point was never reached. Vernon's results for lead and zinc are summarized in Table VII.

TABLE VI.—SEVERE INDOOR CORROSION OF LEAD-PLATED STEEL AT WOOLWICH, ENGLAND.

Coating	0.00005-in. Thick Coating	0.0005-in. Thick Coatng	0.002-in. Thick Coating	Effect of Scratch Through 0.00005 in. Coating
Lead.....	Numerous rust spots	Few faint rust spots	No rust, bluish color	Rusted
Nickel...	Completely covered with rust	Numerous rust spots, greenish spots on nickel	Almost free from rust spots, greenish spots on nickel	Rusted
Copper ..	Almost completely covered with rust	Few faint rust spots	No rust, tarnished	Rusted
Zinc	Almost free from rust	No rust, darkened faint whitish spots	....	Faint rust

Here again the corrosion products of the lead show protective qualities while those of the zinc have no protective value (note the straight-line relation). The rate of corrosion of the lead is again very small relative to that of the zinc.

UNDERWATER TESTS ON SOLID LEAD

No published tests on the behavior of

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[8] Gray and Blum, "Modern Electroplating," *Transactions*, Electrochemical Soc., p. 227 (1942).

[9] Kurrein, *Werkstattstecknik*, Vol. 26, p. 458 (1932).

[10] See also Freitag, *Oberflächentechnik*, Vol. 11, No. 1, p. 5 (1934).

[11] Blum, *et al*, *Transactions*, Electrochemical Soc. Vol. 36, p. 243 (1919).

[12] Vernon, *Transactions*, Faraday Soc., Vol. 23, p. 113 (1927).

lead-plated steel under sea water have been found. However, Friend's[6] work on solid lead and other metals is of some interest. One series of specimens was immersed for 4 yr. in the rapid tidal currents in the Bristol Channel,[13] the time of immersion being 93½ per cent of the total exposure time. The second series was immersed for 3 yr. in the quiet, muddy, and oily waters of the Southampton Docks, England.[14] In both cases the specimens were 2 ft. long, 1⅛ in. in diameter solid rods, held horizontally in wooden frames. One inch on each end of the specimen was set in an "insulating" material, consisting of putty in the Bristol Channel tests and wax or tar in the Southampton Dock tests. Some rather deep pitting on the zinc specimen in the Bristol channel was ascribed to the action of the putty.

TABLE VII.—MILD INDOOR CORROSION OF SOLID METALS AT SOUTH KENSINGTON, ENGLAND.

Time, days	Weight Increment, mg. per sq. dm.	
	Lead	Zinc
30	0.155	0.631
120	0.281	2.46

The results of these tests, recalculated to inch average penetration in 3½ yr. are shown in Table VIII.

Considering our general knowledge of the porosity of lead coatings, these results indicate that lead coatings of the thicknesses covered by the new lead-plating specifications probably would not protect steel very long when immersed even in quiet sea water. Considerably heavier coatings would be required.

LEAD-PLATED STEEL IN CONTACT WITH OTHER METALS

Since the lead plating on steel probably has the same effect as solid lead, the data obtained by Committee B-3[7] appears directly applicable. They are summarized in Table IX.

One may conclude that under no circumstances should lead-plated steel come in contact with aluminum. Generally speaking, it should also be kept away from contact with zinc and zinc-plated parts, although one may expect that a small lead-plated part would not do very great damage to a large zinc-plated part.

Lead-plated steel ordinarily would not cause acceleration of the corrosion of copper, iron, nickel, or tin. As a matter of fact, these metals are protected in the three seacoast atmospheres and in the relatively noncorrosive rural and desert atmospheres. In the industrial atmospheres, the lead may act either way, but in no case is the acceleration of the corrosion of either the lead or the four metals very pronounced. Hence, lead-coated parts could probably be used in contact with these metals with impunity.

TABLE VIII.—UNDERWATER TESTS ON SOLID METALS.

Metal	Average Penetration in 3½ yr., in.	
	Bristol Channel	Southampton Docks
Lead, polished	0.00176	0.00029
Nickel, scaly	0.00115	0.000019
Copper, polished	0.00992	0.00027
Zinc, polished	0.0129	0.00150

PHYSICAL PROPERTIES

The physical properties of lead coatings have considerable bearing on whether such coatings will prove useful in any particular application. A short summary of these properties are, therefore, given here.

The *melting point* of lead is very low, 327.4 C. (621 F.). The boiling point of lead is high, namely, 1613 C. (2935 F.). While lead or lead-bearing *fumes* were not found in certain tests at 520 C.

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[13] *Journal*, Inst. Metals, Vol. 39, p. 111 (1928).
[14] *Journal*, Inst. Metals, Vol. 48, p. 103 (1932).

(970 F.), fumes of lead oxide are formed at a bright red heat. Since lead salts are very *poisonous*, care should be taken to avoid high-temperature applications.

It has been found [15] that while a lead-coated steel sheet can be easily *spot welded* to plain steel, it cannot be spot welded to another lead-coated sheet. One can solder to lead-coated steel but only with some difficulty.

The *electrical conductivity* of lead is about 8.2 per cent of that of silver.

TABLE IX.—PROTECTIVE ACTION OF LEAD IN CONTACT WITH AN EQUAL AREA OF ANOTHER METAL WHEN EXPOSED TO OUTDOOR ATMOSPHERE.

Test Site	Couple					
	Pb/Al	Pb/Cu	Pb/Fe	Pb/Ni	Pb/Sn	Pb/Zn
LaJolla, Calif. (foggy seacoast)	1.01/6.24 —	4.50/0.13 +	2.47/0.52 +	3.31/0.00 +	1.62/0.44 +	4.16/2.10 +
Sandy Hook, N. J. (smoky seacoast)	1.48/6.75 —?	1.67/0.93 +	1.78/0.45 +	1.40/0.43 +	1.52/0.74 +	2.06/2.08 +
Key West, Fla. (seacoast)	0.92/11.00 —	1.06/0.47 +	1.44/0.36 +	1.18/8 +	1.07/0.63 +	1.16/1.43 —?
Pittsburgh, Pa. (industrial)	1.61/7.81 —?	1.18/1.30 —??	1.39/1.10 +?	1.55/1.19 +?	1.23/1.17 +?	1.55/1.67 —??
Altoona, Pa. (industrial)	1.21/4.45 —?	1.25/1.00 +?	1.42/1.01 +?	1.01/0.97 +?	0.96/1.00 —?	1.07/1.99 —
Rochester, N. Y. (industrial)	0.78/3.34 —	1.00/1.42 —?	1.43/1.37 +??	1.21/1.62 —?	0.91/0.91 ?	1.03/1.93 —
New York, N. Y. (industrial near seacoast)	1.06/4.95 —	1.12/1.30 —??	1.06/0.98 +	0.93/0.89 +??	0.85/1.06 —	1.16/1.68 —
State College, Pa. (rural)	1.22/8.50 —?	1.28/0.38 +	1.57/0.62 +	1.25/0.00 +	1.18/0.08 +	1.37/1.74 —?
Phoenix, Ariz. (desert)	1.29/6.34 —?	1.19/0.80 +	1.24/0.67 +	0.85/0.00 +?	0.90/0.22 +?	1.07/2.30 —

NOTE.—The ratios and + and − signs have the same meaning as in Table V.

Lead is used on certain *electrical contacts*, for example, in connection with storage batteries. While the lead corrosion products are poor conductors, they are fairly easily penetrated because of the softness of the underlying lead, and contact is established by sharp points.

Next to thallium, lead is the softest of all the heavy metals. Brinell *hardness* as low as 3 kg. per mm. has been found at room temperature. The plated coatings are somewhat harder. Lead hardens on *cold working* but self-anneals at room temperature, the rate of softening being extremely rapid after severe cold working. Because of its softness, a lead coating is easily cut through by sharp points. Where lead-plated parts are handled roughly, for example, in the assembly of structural parts, an extra thickness may have to be provided for.

The softness of lead, however, has its advantages, for example, in the case of lead-plated gaskets and packing rings. Bolts and nuts do not require plating to precise tolerances. When hit with blunt objects the lead flows but does not crack.

The *tensile strength* of lead is very low, from about 1800 to 3000 psi., depending on the amount of cold work and the rate of testing. In spite of this, lead-plated sheets are easily bent without cracking of the coating. The reason is that the percentage *elongation* is very high, some 60 per cent on fast loading and much higher on slow loading.

Lead plating in fluoborate solutions does not cause embrittlement of the base metal although, of course, pickling preceding such plating may embrittle the steel as usual. This makes lead a suitable coating on springs and parts subject to vibratory stresses.

The *color* of lead is bluish-gray. It is much darker than most white metals and is ordinarily not considered of much decorative value.

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[15] Private communication from C. E. Heussner, Chrysler Engineering Laboratories, Detroit, Mich.

The *specific gravity* of lead is about 11.35. One cubic foot weighs about 706 lb., and one cubic inch about 6.53 oz.

A 0.0001-in. thick coating weighs 0.094 oz. per sq. ft. = 0.00065 oz. per sq. in. = 0.0185 g. per sq. in. = 2.67 g. per sq. ft.

USES FOR ELECTRODEPOSITED LEAD COATINGS ON STEEL

Until the outbreak of the present war, lead plating on steel in this country was largely limited to a few uses in which advantage was taken of the resistance of lead to chemical corrosion. Blum, *et al*,[11] cites such uses as storage battery fittings, boosters, adapters, and linings of gas shells as being in actual use, and gas heater flues, brine tanks for refrigeration, and linings for chemical apparatus as proposed. Thicknesses of 0.003 to 0.005 in. of lead are suggested as being substantially nonporous. Gray and Blum[8] state that lead plated nuts and bolts usually have a thickness of 0.0005 to 0.001 in. and that most ferrous metals are plated with a thickness of 0.0005 to 0.008 in. depending on the subsequent exposure of the plate.

Electrolytic lead coatings appear to have been used to a considerably larger extent in Continental Europe. The Schlotter process which makes use of a lead phenosulfonate bath is being employed in Germany on a variety of parts. Ecker[16] shows various types of coil springs, gas mask parts, steam water heaters, and packing rings for same being lead plated by this process. He advocates the use of steel sheet, lead-plated to a thickness of 0.00052 in. for roofing sheet, storage tanks for gasoline and oil, automotive gasoline tanks, and ice containers. According to American experience[15] somewhat heavier coatings, a minimum thickness of 0.00075 in., are required on the bottom of automotive gasoline tanks for most satisfactory results.

Ecker,[16] Kurrein[9], and Freitag[10] mention the wide-spread use of lead coatings on steel parts such as structural members used in the building of railroad stations, tunnels, and bridges, and on railroad screws, bolts, and nails. The German State Railways specify exceedingly thick coatings on structural parts which have to stand up in this very severe service: 0.012 in. providing an expected life of 10 to 20 yr. against 3 to 5 yr. for the best paint coatings.

Hay[17] discusses uses for lead plating in Switzerland and cites: supports for electric lines, channel irons for cables, earthpoles for grounding high tension lines at power stations; structural parts for factories, foundries, railroad stations, etc., and chemical plants. The thickness of the lead coatings vary depending on the conditions of exposure, from 0.008 to 0.12 in. The latter figure probably refers to strictly chemical uses.

Gray and Blum[8] state that nuts and bolts usually have a thickness of 0.005 to 0.001 in.

Acknowledgments:

The kind assistance of C. E. Heussner of the Chrysler Corp. in making available some test results and experiences had by his company, and of Thor H. Westby Technical Laboratories, Sears & Roebuck Co., and Chairman of Subgroup on Lead Coatings on Steel of Subcommittee I of Committeee B-8 in gathering some of the data presented here, is most gratefully acknowledged.

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[16] Ecker, "Problems of the Metal Industry," Evalenko Verlag, New York, N. Y. and Berlin, p. 79 (1930).

[17] Hay, *The Metallurgist*, Vol. 11, p. 23 (1937).

DISCUSSION

MR. SAM TOUR.[1]—I should like to compliment Mr. Soderberg on his very excellent paper, and at the same time sound a word of caution with respect to some of the things in the paper.

Of particular interest are the references to reports of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys. Committee B-3 has spent many years collecting and publishing factual data and warning against drawing too many conclusions from these data, and warning against trying to weave in between the data explanations of why the results were as reported. I want to repeat that warning. In selecting certain of these data to incorporate into this paper, Mr. Soderberg has attempted to read into the data an explanation of why the data are as they are. For 23 yr. we have been trying to collect data and find some of the answers. So-called rural, suburban, and urban atmospheres in England act in their peculiar ways. In the United States, Committee B-3 calls certain corrosion test locations rural, suburban, or urban. The atmosphere called rural in the United States is not necessarily the same as one called rural over in England. It should not be assumed or said that a given set of metals will act the same some place in the United States as they have apparently acted some place in England. In all the years of study in Committee B-3 we have not been able to pin down corrosion and say, "it happens in this way."

It is necessary to sound a word of caution about how far one can go in drawing conclusions from corrosion data. No matter how conscientiously the corrosion work may have been carried out there is great danger in jumping to conclusions.

It is not meant that lead-coated steel is not perfectly satisfactory for certain uses. I wish to point out that Committee B-3 results on galvanic couples of steel with lead and aluminum exposed in Arizona or Pennsylvania are not proof of what results would be under other conditions.

Mr. Soderberg referred to the hazard of lead poisoning due to lead fumes from heating of lead. I wish to emphasize the hazard of lead poisoning due to the handling of lead. Recently I was astonished to see an item in one of the technical journals advocating lead-coated steel for Army mess kits. I certainly do not want my boy to be forced to eat food from lead-coated plates. Lead is poison whether it is inhaled as fumes from heated lead or whether it is taken into the stomach with food. It has been proposed that lead-coated steel be used for steel cartridge cases. Ammunition must be handled with bare hands. After handling lead-coated ammunition and before eating a thorough washing of the hands would be mandatory if lead poisoning were to be avoided. Soap and water, time and facilities for such washing are not always available at the "front." Lead-coated ammunition would be an unnecessary hazard imposed on our fighting forces.

MR. C. D. HOCKER.[2]—Like Mr. Tour,

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[1] President, Sam Tour and Co., Inc., New York, N.Y.

[2] Plant Products Engineer, Bell Telephone Laboratories, Inc., Murray Hill, N. J.

I, too, would like to express my appreciation to Mr. Soderberg for what I think is a very excellent review of a paper on lead coatings.

There does occur on top of page 565 one statement which I should like to remark on in relation to the findings of Committee A-5 on Corrosion of Iron and Steel, and that is the statement: "In a strictly industrial atmosphere lead does not offer much, if any, electrochemical protection and may actually cause acceleration of the corrosion of the steel." That statement may be true. It is interesting, I think, however, to recite the findings of Committee A-5 over the past 20 yr. in its observation of lead-coated hardware that has been exposed outdoors, and again the observations of lead-coated wire exposed outdoors.

In the case of hardware which has been exposed outdoors for 15 yr. or more, even in a severely industrial atmosphere like Pittsburgh and Altoona, there has been a very good protection of the underlying iron. Whether that protection is electrochemical or whether it is due to some mechanism such as the plugging of pores by corrosion products, or whatever it may be due to, it is very obvious that there is a striking protection of the underlying iron.

It is very obvious when you look at the many samples that have been outdoors that all of them showed evidence of pin-holes. These became evident very shortly after the specimens were exposed, but the corrosion has not spread from those pin-holes and resulted in an over-all rust scale of the underlying metal.

It is also rather striking in a few places where opportunity has been afforded to make the observation—places where an occasional lead-coated sample has fallen off the rack and has had contact for a few weeks or months with the ground—that the fallen specimens are much more corroded than their companion samples that have stayed up on the racks.

As stated briefly in the current Report of A-5, the same is true of wire specimens, whether they be exposed in industrial locations like Pittsburgh or in rural atmospheres like Manhattan, Kans. Wire specimens which were coated with lead have not shown any loss in strength when they have been brought in for tests, even though in every location they did show evidence of rust stain within a few weeks after they were exposed.

MR. C. E. HEUSSNER.[3]—I cannot quite agree with Mr. Tour's discussion of poisoning by lead. We all agree that lead fumes are very poisonous but there is a great deal of discussion as to the amount of poisoning produced by the handling of lead. I doubt whether lead is absorbed in the body in this manner. We know that for years people have worn lead crucifixes and have never been poisoned, and, in general, it is only the fumes of lead that are absorbed internally that become dangerous.

MR. K. GUSTAF SODERBERG[4] (*author's closure by letter*).—I am grateful for the comments made. In reply to Mr. Tour, I should like to mention that if the results of an exposure test at a certain location, for example, in Pittsburgh, did apply to that location only, there would be little gained in making the exposure test at all. At a time when only limited information is at hand, it appears more desirable to utilize the available data to the utmost and take some chances of going too far, than to leave the choice of coatings to chance entirely or to judgment based on unreliable accelerated tests.

All of Mr. Hocker's references deal with heavy lead coatings in which the pores appear to become sealed off in time. The thin coatings observed in London apparently did not recover by such a process.

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[3] Materials Engineer, Chrysler Corp., Detroit, Mich.

[4] Technical Director, The Udylite Corp., Detroit, Mich.

LEAD-ALLOY COATED COPPER WIRE FOR ELECTRICAL CONDUCTORS

By C. J. Snyder[1]

Synopsis

An effort to save tin for the more essential war needs has led to the substitution of lead alloys for pure tin when coating copper wire for protection against rubber compounds.

The results obtained when wire was coated in baths of three different binary tin-lead alloys are discussed. It was shown that when a bath of 10 to 66 per cent tin, remainder lead, was used, the coatings were segregated with the tin concentrated near the copper and the lead concentrated on the surface. Coatings from a 5 per cent tin-lead bath were not segregated. All these coatings, however, were relatively "sticky" in stranding and insulating operations.

A new lead alloy containing small amounts of tin, cadmium, and antimony was developed for this application. This new alloy-coated wire more nearly approaches tinned wire in physical properties than any other tried. This alloy can be applied with standard equipment, and a method of control to keep the coating uniform was worked out. Data show that substitution of this alloy saved 87 per cent of the former tin consumption.

The war in the Pacific has created a shortage of tin. It was, therefore, necessary to restrict the use of tin to its more essential applications. The coating of copper wire with pure tin consumed large quantities of tin in normal times. Much of the wire used for electrical conductors was tinned to facilitate the soldering of joints, to protect the copper against corrosion by certain ingredients present in rubber insulation, and to protect the rubber insulation against deterioration by the copper. Alloy coatings had been substituted to some extent in the past but had never become universal because of the very desirable properties inherent in coatings of pure tin. Under present regulations,[2] an alloy containing not over 12 per cent tin must be substituted for pure tin for coating copper wire except on materials for combat use. A lead alloy was the logical choice as a substitute because lead is plentiful.

A blanket substitution of lead alloy for tin coatings, however, created many problems, both as regards the nature and application of the coating and regarding usability of the coated wire. The main requirements of a coating as set forth in specifications are composition, continuity, adhesion, and smoothness. These requirements can be met, after some manufacturing experience, by using an acceptable low-tin lead alloy. The resulting product may not be a usable substitute, however, because of other unnamed requirements inherent in tin coatings and not present in alloy coatings. The coating must be capable of being soldered easily, must possess good resistance to abrasion, exceptionally good antifrictional properties, and a certain resistance to corrosion by sulfur and rubber chemicals, if the alloy-coated wire is to be successfully and completely substituted in all the various types of twisting, stranding, and cabling machinery and various models of insulating machines without retarding the war output. It is probable that lead-alloy coated wire would have, to a large extent, replaced tinned wire many years ago, if it were not for some of the mechanical difficulties in stranding and insulating the wire.

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[1] Engineer-in-Charge, Metallurgical Laboratory, Anaconda Wire and Cable Co., Hastings-on-Hudson, N. Y.

[2] WPB Order M-43, amended January 9, 1943.

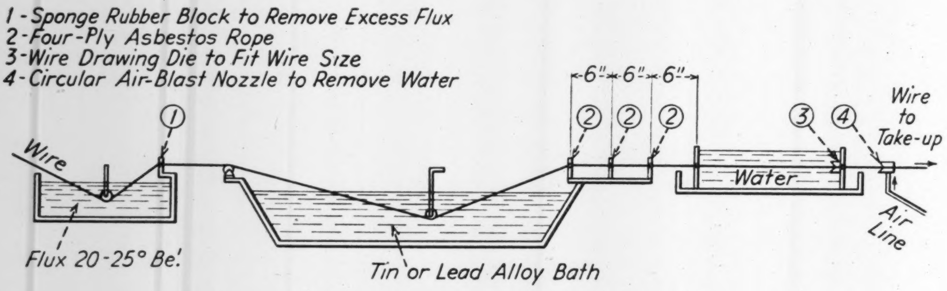
1 - Sponge Rubber Block to Remove Excess Flux
2 - Four-Ply Asbestos Rope
3 - Wire Drawing Die to Fit Wire Size
4 - Circular Air-Blast Nozzle to Remove Water
Wire
1
Flux 20-25° Be'.
Tin or Lead Alloy Bath
6"
6"
6"
2
2
2
Water
3
4
Wire to Take-up
Air Line

FIG. 1.—Schematic Diagram of Wire Tinning Process.

GENERAL TINNING PROCESS

Before proceeding with the experimental data, a brief description of the coating process in general use, that is, the hot-dip process, will be beneficial to the understanding of the data. The wire, which must be clean and bright from previous operations, is passed, single-strand fashion, through a flux bath or in some way wetted with a thin film of a suitable fluxing agent after which it passes rapidly through a molten bath of tin. A temperature of 525 to 560 F. is used. As the wire emerges from the bath it passes through suitable wiping devices first to remove the majority of the molten metal and then to smooth the surface. The wire is immediately cooled, usually by passing it through a circulating water bath, and then dried before spooling. (See Fig. 1.) The details of the practice may vary considerably from mill to mill and for different sizes of wire.

The process for applying a lead alloy is, in general, the same as for applying a tin coating. Closer control of temperatures, wire speed, and wiping conditions is necessary, however; and, in addition, the composition of the alloy must be kept uniform. The alloy bath is maintained at a higher temperature than pure tin and should be protected from the air. Bath temperatures of 650 to 810 F. have been used successfully, depending on wire size and other conditions, and the selected temperature should be maintained within ±10 F. Excess flux should be prevented from entering the bath. The wire speed should be comparatively rapid, as for example, 600 ft. per min. for an 0.063-in. diameter wire or 850 ft. per min. for an 0.013-in. diameter wire. A number of forms of heat-resisting wipers have been used for removing the excess alloy. Probably the most common of these is one or more 4-ply asbestos cords tightly looped three or four times around the wire. A pair of blocks of special heat-resisting rubber compressed around the wire makes an effective smoothing wiper. After water cooling, the wire must be thoroughly dried before spooling to prevent corrosion of the lead coating.

During continuous operation, the lead-alloy bath becomes rapidly depleted of some of the alloying elements, notably tin, and routine control to replace the lost elements is necessary to insure uniform operating conditions. This, in general, can be done by using as replacement metal for the bath, an alloy that is relatively richer in the depleted elements than is normally maintained.

BINARY TIN-LEAD COATINGS

Before presenting the data regarding the alloy now successfully in production for the past 16 months, the results of some previous operations when various binary lead-tin alloys were used will be discussed.

A tinning machine containing 4410 lb. of alloy made up to an exact 10.0 per cent tin content and containing 0.04 per cent copper was operated for three 8-hr. days and a total of 12,970 lb. of Nos. 12 and 14 A.w.g. wire was coated. At the end of this time 214 lb. of alloy were used up and the bath analyzed 8.10 per cent tin and 0.22 per cent copper. Therefore, 101 lb. of tin *versus* 113 lb. of lead was used up and the bath picked up 9 lb. of copper. Analyses of the coatings from 28 wires showed the coatings varied from 7.7 per cent to 29.1 per cent tin, remainder lead. More data were obtained at a later date when

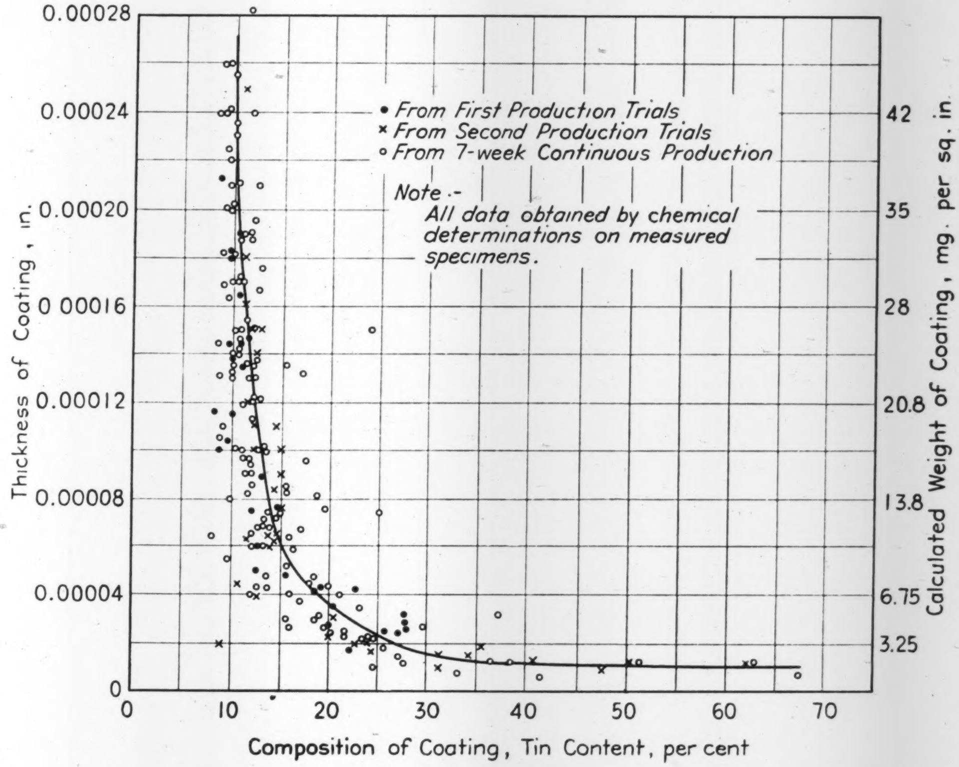

FIG. 2.—Relationship of Composition to Weight of Coating on Copper Wires Coated in Bath of 10 per cent Tin, 90 per cent Lead Alloy.

an additional 20,000 lb. of wire was coated. The alloy bath was again made up to a 10.0 per cent tin-lead composition and was tried at various temperatures between 625 and 685 F. Analyses of the coatings of 38 new samples showed variations in tin content in the coatings of 8.8 per cent to 86.3 per cent tin. A constant relationship between the coating thickness and the tin content of the coating was noted, and the curve shown in Fig. 2 was plotted from the 66 samples analyzed during these two production periods. The data indicated that the alloy was segregated in the coating, the tin concentrating near the copper and the lead on the surface.

Additional data were obtained when a coarse-wire tinning machine, taking wire 0.048 to 0.128 in. in diameter, was operated continuously for 7 weeks, during which time 199,000 lb. of wire was coated. The pot was fed with 10 per cent tin alloy, 2465 lb. of which was consumed, and was maintained between 700 and 775 F., a higher temperature than before. At the end of this time the bath contained only 7.40 per cent tin and had picked up 0.44 per cent copper. Many different types and arrangements of wipers were used in an effort to overcome the variations and the relatively thick coatings previously obtained. Analyses of 163 samples showed the tin content in the coatings varied from 7.9 per cent to 67.5 per cent and the same relationship of coating thickness to tin content in the coatings previously noted was again obtained. These data are plotted as circles in Fig. 2.

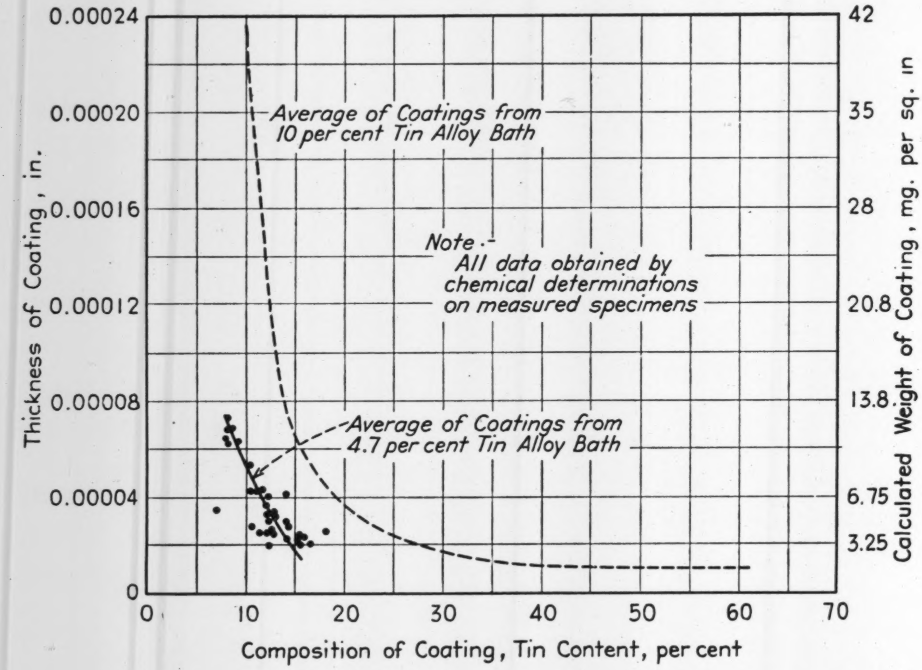

FIG. 3.—Relationship of Composition to Thickness of Coating on Copper Wires Coated in Bath of 4.7 per cent Tin, 95.3 per cent Lead.

In addition to these variations in

coatings, the 10 per cent tin lead alloy was more difficult than tin to apply smoothly and the coated wire had comparatively poor resistance to abrasion and poor antifrictional properties, commonly spoken of as "sticky," when stranded or when used in continuous-vulcanizing rubber extrusion equipment.

Because of previous experience, coating wire in a bath of lower tin content as did the 10 per cent alloy bath. Analyses of 34 samples of wire showed the tin content in the coatings varied from 6.9 to 18.3 per cent. The constant relationship between tin content and coating thickness, shown in Fig. 2, did not exist when a 5 per cent tin bath was used. These coatings were comparatively uniform in composition and weight as shown in Fig. 3. The alloy was also

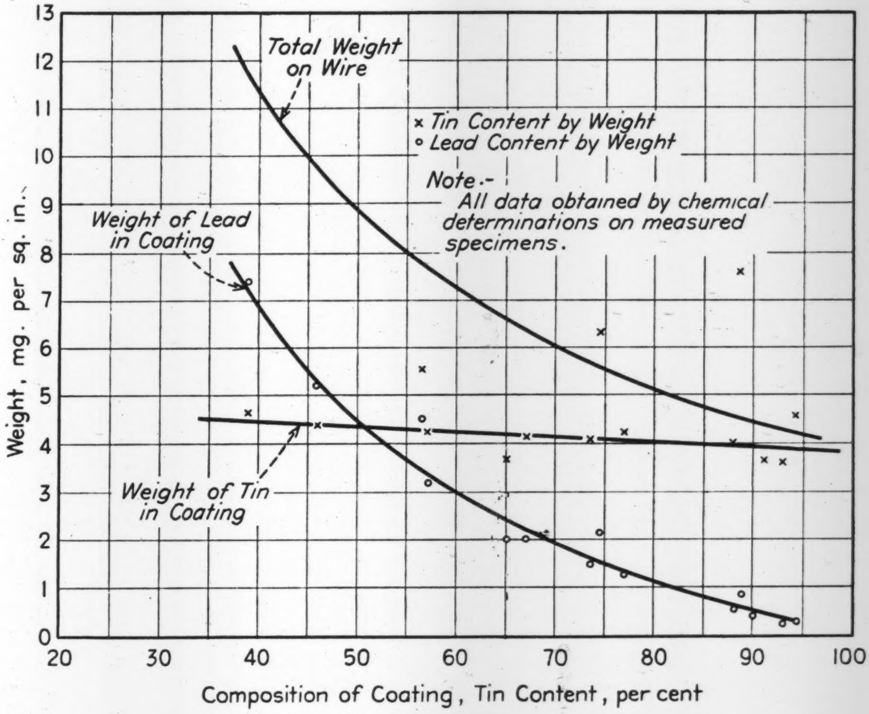

FIG. 4.—Relationship of Composition to Weight of Coating on Copper Wires Coated in Bath of 66 per cent Tin, 32 per cent Lead.

was tried. A coarse-wire tinning machine was operated continuously for 6 days with the alloy made up to contain only 4.7 per cent tin. The alloy bath was maintained at temperatures between 740 and 780 F. Approximately 30,000 lb. of 0.064-in. diameter wire was coated after which the bath analyzed 3.38 per cent tin and 0.06 per cent copper. This low-tin alloy did not pick up copper more easily applied smoothly and rapidly in production. The usability of the coated wire was restricted, however, because the coating was soft and "sticky" so that the mill had much difficulty when stranding and insulating the wire, the turns of wire sticking together on the reels.

When the crisis in tin supply first became apparent, an effort was made to save some tin and at the same time have a wire which could be readily substituted for tinned wire by coating wire in a bath that analyzed 66.4 per cent tin, 32.0 per cent lead, and 1.6 per cent copper. The alloy was maintained at a temperature of 563 F. and was easily applied to yield a smooth coating. Analyses of 14 samples collected from 20,000 lb. of various sizes of wire showed the tin content in the coatings varied from 38.9 per cent to 94.3 per cent. These analyses further indicated that the weight of tin per square inch of wire surface in the coatings was about constant on all samples, while the total weight of coating increased as the lead content increased, thereby indicating extreme segregation. These data are plotted in Fig. 4. The use of a high-tin lead alloy, therefore, saved very little tin and the coating, although comparatively resistant to abrasion, was "sticky" as compared with tinned wire.

The above trials indicated that the only tin-lead alloy that produced a reasonably uniform thin smooth coating and could be depended upon to save appreciable amounts of tin was one containing less than 5 per cent of tin. The wire coated with this alloy was not easily substituted for tinned wire, however, because special handling during stranding and insulating was necessary and production was greatly impeded.

NEW ALLOY COATING

A new alloy[3] was, therefore, developed for this application. Many lead-base alloys were tried but it was found that lead to which small amounts of cadmium, tin, and antimony were added was the only one of those tried which approached the desired properties. These three elements were added in suitable proportions to give their maximum hardening effect with the tin content controlled at 2 per cent tin in the bath. It was further found that a bath of this alloy in continuous operation did not pick up more than 0.07 per cent copper.

After the initial trial period, this alloy could be applied in smooth, bright coatings without difficulty. During a period of 6 weeks of gradual change-over, this alloy was substituted in place of pure tin for coating all wire 0.040-in. in diameter and larger, amounting to approximately 3,000,000 lb. per month in one mill without seriously interrupting production schedules of bare and insulated wires and cables. Later the alloy was substituted for coating all sizes of wire. It was more difficult to get started on the small sizes, but during a period of 3 months, all serious difficulties were eliminated by careful control and thereafter standard production schedules were maintained. This change-over not only involved the coating of the bare wire with alloy but also the stranding of the coated wires into cables of various types and the application of rubber, plastic, and other insulating materials to the single and stranded conductors.

After 16 months of continuous and almost exclusive use, during which time some 50,000,000 lb. of wire of all sizes has been coated, stranded, and insulated, it can be said that wire coated with this new alloy has properties approaching those of tinned wire.

Analyses of 47 representative samples of various sizes of wire from production showed that the tin content in the coatings varied from 0.98 per cent to 16.8 per cent. The coating tests showed only slight segregation as indicated by the relationship between the weight of coating and the composition of coating. These data are plotted in Fig. 5. The weight of coating tended to be relatively high, but this seemed to depend on the effectiveness of the wiping devices rather than on the coating composition. The average composition of the coatings of all samples analyzed to date was as follows:

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[3] Patent applied for.

AVERAGE ANALYSES OF COATING FROM NEW ALLOY

Tin, per cent	5.23
Cadmium, per cent	1.17
Antimony, per cent	0.30
Lead, per cent	93.30

Coated Copper Wire for Electrical Purposes (ES-1a)[4] (issued June 18, 1942). It was rare indeed that the new lead-alloy coated wire did not pass a 30-min. immersion. In this respect, the alloy coated wire surpasses tinned wires which occasionally fail in this test. Wire coated with the new alloy will often meet the continuity of coating tests after it has been cold drawn to meet the tensile requirements of the specifications for medium-hard and hard-tinned wire.

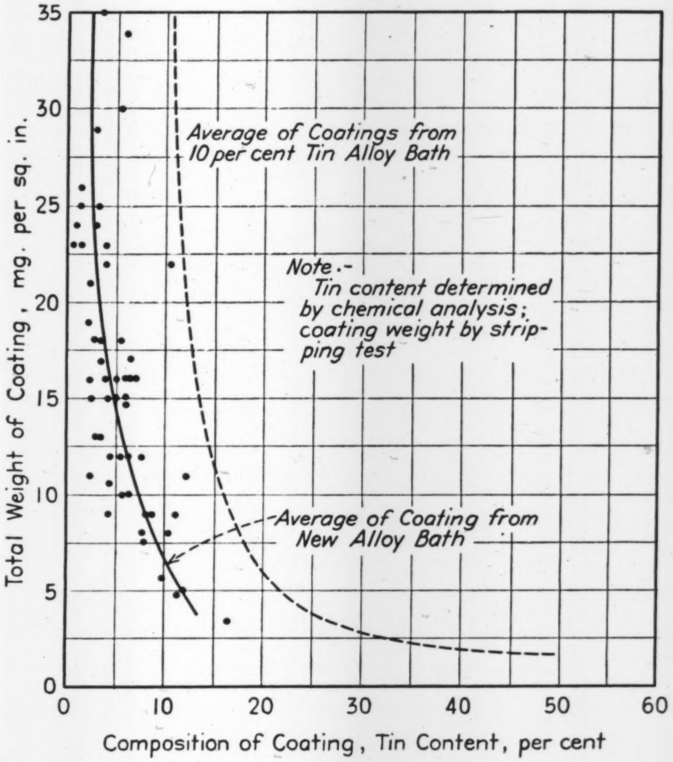

FIG. 5.—Relationship of Composition to Weight of Coating on Copper Wires Coated in Bath of New Cadmium-Tin-Lead Alloy.

Continuity of Coating:

The continuity of coating of this wire can readily be tested by the ammonium persulfate test as described in Section 7 of the A.S.T.M. Emergency Specifications for Lead-Coated and Lead-Alloy-

Soldering:

No unusual difficulties have been experienced when making soldered joints involving this new alloy-coated wire. All ordinary types of electrical joints, using typical wire and cable sizes, have been satisfactorily soldered in the usual way with prewar solders. When joints were soldered with some of the new low-tin war solders, the results were not so satisfactory. The same difficulties were encountered, however, when applying the low-tin solders to joints between tinned wires. It therefore appears that the trade has undergone at about the same time changes in both solder compositions and wire coatings. This may have caused some difficulties which were attributed to the wire coating but which probably should have been attributed to the solder. When difficulties are experienced under present conditions, it may be well to try a stearin or other flux especially suited to lead alloys and to try a temperature slightly higher than customary to make up for the lower tin content of the solder.

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[4] 1942 Book of A.S.T.M. Standards, Part I, p. 943.

COATING PROCESS

The machines formerly used for tinning are being used for alloy coating. About the only changes necessary were to equip the pots with pyrometers for higher temperatures and to move the wipers to a location where they were least affected by the heat. Below are listed seven points which are necessarily followed for successful operation:

1. The lead alloy should be maintained at a temperature of 740 to 800 F., depending on the size of the wire.
2. The lead alloy must *not* be overheated or its composition will change.
3. The lead alloy should be covered at all times with a generous layer of impregnated charcoal.
4. The wire should be clean and unoxidized. When passing through the flux, it must be wetted and the excess flux removed so that there will be very little boiling in the lead pot.
5. Suitable wipers must be used to remove the excess alloy from the wire before cooling. These may consist of asbestos cords, special high-temperature rubber, dies, or other customary units capable of withstanding the temperatures needed.

TABLE I.—UNIFORMITY OF ALLOY IN ONE WIRE-COATING MACHINE.

Obtained under mill conditions by using hardness control.

Date	Brinell Hardness Number	Metal Added, lb.		Composition of Pot, per cent			Wire Production, lb.	Major Wire Size Diameter, in.
		Special Intermediate Alloy	Make-up Alloy	Tin	Cadmium	Lead (by Difference)		
8/24/42	8.64	20	600	1.67	0.56	97.77	32 909	0.0612
8/25/42	9.17	10	400	1.89	0.67	97.44	24 463	0.051
8/26/42	9.28	0	400	1.97	0.65	97.38	37 586	0.0651
8/27/42	8.74	20	400	1.96	0.61	97.43	27 866	0.1055
8/28/42	9.57	0	200	1.99	0.70	97.31	27 632	0.0772
8/29/42	8.90	10	0	1.82	0.51	97.67	4 412	0.045
8/31/42	9.06	10	400	1.80	0.73	97.47	35 173	0.091
9/ 1/42	9.22	0	300	2.01	0.75	97.24	18 398	0.045
9/ 2/42	9.34	0	300	2.00	0.70	97.30	31 890	0.045
9/ 3/42	8.74	20	100	1.97	0.61	97.42	25 924	0.0772
9/ 4/42	8.90	30	400	2.08	0.74	97.18	29 137	0.0822
9/ 6/42	no test	0	0	no test	...	...	12 300	0.057
9/ 7/42	8.74	20	300	1.71	0.65	97.64	29 700	0.045
9/ 8/42	9.06	10	400	2.01	0.77	97.22	32 587	0.0772
9/ 9/42	9.34	0	300	2.13	0.76	97.11	36 145	0.057
9/10/42	8.85	10	300	2.12	0.70	97.18	34 871	0.0772
9/11/42	8.64	20	300	2.12	0.69	97.19	39 613	0.0772
9/12/42	9.06	10	300	2.25	0.79	96.96	33 057	0.057
9/14/42	9.64	0	400	2.22	0.82	96.96	26 695	0.0612
9/15/42	8.64	20	400	2.04	0.70	97.26	25 570	0.057
9/16/42	9.06	10	300	2.18	0.78	97.04	31 376	0.0486
9/17/42	9.22	10	200	2.10	0.76	97.14	19 002	0.0612
9/18/42	8.95	10	200	2.06	0.75	97.19	28 554	0.0772
9/19/42	9.17	10	100	2.05	0.76	97.19	30 320	0.0612

Summary for Four Weeks' Production:

Pounds of wire coated = 675,180

	Tin	Cadmium	Antimony	Lead	Total
Metal consumed, lb.	528	326	26	7765	8645
Per cent of total.	6.10	3.77	0.31	89.82	

Approximate weight of tin needed to coat the same wire = 4000 lb.
Amount of tin saved by using alloy = 3472 lb.
Amount of tin saved by using alloy = 86.9 per cent.

6. *All* water must be removed from the wire with an air nozzle or other suitable means before spooling, or the coating will corrode rapidly.
7. The wire should be run at normal tinning speeds, the faster the better, as long as a consistently adherent coating is obtained.

CONTROL OF ALLOY COMPOSITION[5]

The working elements, tin and cadmium, of the new alloy are depleted slowly by use. The standard pot composition can be normally maintained by using alloy pig metal relatively richer in tin and cadmium for replacement of the metal used from the pot. It was found by experience that a make-up alloy containing approximately 2½ times the required content of these metals was most effective for this purpose.

The bath in each coating machine should be tested for composition daily. In lieu of chemical analyses, which are time consuming, a Brinell hardness test is made and the resulting values compared with those in a standardized chart. It was found that when the Brinell number fell below a set value the bath usually needed more cadmium, which was added in the form of small coupons of special intermediate alloy. The data in Table I show an example of the uniform composition obtained by this system of control.

SAVINGS OF TIN

The data at the bottom of Table I show the amount of tin saved by substituting the new lead alloy for tin. This saving was reliably calculated at approximately 87 per cent of the previous tin consumption. One mill of the author's company has therefore saved approximately 200,000 lb. of tin in the past 12 months.

CONCLUSIONS

In conclusion, therefore, the development and application of a new lead-alloy coated copper wire has been shown which has proved useful to the war program. The alloy can be applied by the conventional tinning machines.

The use of this alloy has saved 87 per cent of the former tin requirements where substituted.

The new wire has a smooth continuous coating which has comparatively good resistance to abrasion and antifrictional properties so that it can be stranded and insulated by most conventional machinery without loss of production.

A simple method of control has been worked out to keep the wire coating uniform from day to day.

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[5] Patent applied for.

DISCUSSION

MR. W. R. HIBBARD[1] (*presented in written form*).—Mr. Snyder's paper is of particular interest to manufacturers of copper wire, and I appreciated an opportunity to study it. For years we have been coating wire with various solder mixtures instead of pure tin. The most common solder was one containing 90 per cent lead and 10 per cent tin. Since we have used this alloy in larger quantities for all of our production, we have found that the addition of small amounts of antimony help to secure a better and smoother coating. We have now standardized on this coating to meet the requirements of A.S.T.M. Emergency Specifications for Lead-Coated and Lead-Alloy-Coated Copper Wire for Electrical Purposes (ES - 1a).[2]

MR. A. BELLIS.[3]—I should like to ask Mr. Snyder whether he has made any investigation of the effect of antimony coating in so far as rubber insulation is concerned where sulfur curing agents are used.

MR. C. J. SNYDER.[4]—No investigation of the effect of the new alloy coating on rubber insulation has been made. The antimony content in this coating always runs less than 0.50 per cent and is therefore present in smaller amount than are the other constituents.

MR. BELLIS.—In connection with the feature of the bending test, have you run bending tests along with the other investigation on this type of coating?

MR. SNYDER.—Bending tests of various types have been made on samples of the commercially coated wire. No difficulty with flakiness or poor adhesion was experienced when wrapping the wire around its own diameter. We have also cold drawn the coated wires, and in some cases we were able to meet the tensile requirements of hard-drawn wire and in addition meet the continuity test required in ASTM Emergency Specifications ES-1a.[2]

MR. SNYDER (*author's closure, by letter*).—The remarks presented by Mr. Hibbard are appreciated. It was noticed, when experimenting with the addition of various elements to lead-base coating alloy, that small amounts of antimony had the effect of giving a smoother coating as stated by Mr. Hibbard. The antimony did not supply the additional toughness that we needed for stranding the wire. Brinell hardness tests on chill cast bars of alloy showed that the antimony between 0 and $\frac{1}{2}$ per cent had little effect on the hardness of the alloy. It was only by the addition of cadmium that the desired toughness and hardness of the coating alloy was obtained.

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[1] Assistant Metallurgist, American Brass Co., Waterbury, Conn.

[2] 1942 Book of A.S.T.M. Standards, Part I, p. 943.

[3] Chief Electrical Engineer, John A. Roebling's Sons Co., Trenton, N. J.

[4] Engineer-in-Charge, Metallurgical Laboratory, Anaconda Wire and Cable Co., Hastings-on-Hudson, N. Y.

EXPERIMENTS ON PLASTIC BENDING FOR ALUMINUM ALLOY 17S-T

By Joseph Marin[1] and F. D. Cotterman[2]

Synopsis

This investigation deals with an experimental study of aluminum alloy 17S-T when subjected to pure bending in the plastic range. Plastic strains and deflections were measured for beams of rectangular cross-section having different ratios of width to depth of specimen. To interpret these test results, theories were developed based on simple tension and compression stress-strain relations. These theories were based on approximations of the simple tension and compression diagrams and give the theoretical strain-moment or deflection-moment relationships for members in bending. A comparison between these theoretical values and the actual ones shows that, within the limits of the variables considered, deflection-moment and strain-moment diagrams in plastic bending can be satisfactorily predicted by the theory developed. A new approximate method is also given for defining the yield strength in bending. This method is correlated to the A.S.T.M. offset method used for tension tests.

Statement of Problem

In structural and machine design, an effort is usually made to maintain stress values that will prevent yielding of the material. In some cases, however, yielding in parts of constructions cannot be prevented, and in others yielding may be permitted without objectionable results. Some information is available for stress analysis and design in the plastic range (1).[3] Little experimental data, except perhaps for mild steel, have been obtained to give sufficient confidence in the use of the theories developed. This paper is an attempt to contribute some test data on the particular problem of plastic bending and to interpret such results. This is done by showing how theoretical load-deflection relations based on simple tension and compression diagrams compare with test data on specimens subjected to plastic bending.

Previous Investigations

One of the first investigations of plastic bending was made by Considere (2) who made tests on steel beams loaded in the plastic range. No interpretation of the data based on tension and compression stress-strain diagrams, however, was made. Kennedy (3) tested 0.29 per cent carbon steel beams of various cross-sections subjected to pure bending. He found that the yield stress in bending based on deflection in beams of rectangular cross-section was 1.46 times as large as in pure tension. For steel, in which the plastic part of the stress-strain diagram is essentially horizontal, theoretical stress-moment and deflection-moment relationships were obtained by Nishihara (4). These relations were checked by tests on beams of various cross-sections. Another investigation on steel bars having a horizontal stress-strain variation in the plastic range was made by Muls (5). Test results on steel beams in plastic bending were more recently reported by Kazinczy (6) and and Leloup (7).

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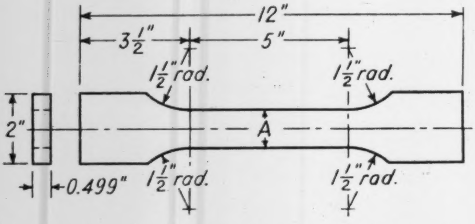
[1] Professor of Engineering Mechanics, The Pennsylvania State College, State College, Pa.

[2] Research Engineer, Crane Co., Chicago, Ill.

[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 594.



Specimen	Depth A, in.	Width, in.
No. 1	0.754	0.499
No. 2	1.000	0.499
No. 3	1.247	0.499
No. 4	1.495	0.499
No. 5	1.758	0.499

FIG. 1.—Dimensions of Test Specimens.

TESTS ON ALUMINUM ALLOY 17S-T IN PLASTIC BENDING

Material and Test Specimens:

The material tested was a wrought aluminum alloy, designated commercially as 17S-T. The nominal composition (8) is 4.0 per cent copper, 0.5 per cent manganese, 0.5 per cent magnesium, and the remainder aluminum and normal impurities. Nominal physical properties (8), obtained by proper heat treatment and aging, are:

Yield point, tension and compression (0.2 per cent offset), psi	37 000
Tensile strength, psi	60 000
Elongation in 2 in., per cent	22
Brinell hardness number	100
Yield point in shear (0.2 per cent offset), psi	22 000
Ultimate strength in shear, psi	36 000
Modulus of elasticity, psi	10 300 000

Specimens were milled from nominal 2 by $\frac{1}{2}$-in. bars as shown in Fig. 1. The specimens were 12 in. long and were provided with a reduced section having a 5-in. gage length.

Test Apparatus:

A diagram of the test apparatus used is shown in Fig. 2, and photographs showing other details are given in Figs. 3 and 4. The ends of the specimen were clamped by the holders *A* and *B* using setscrews and steel blocks as shown. Rollers at *R* and *S* support the holders and a loading bar *D* is connected to these holders by the hangers *C* and *E*. By means of the lever *F* and the weights *W*, a load could be applied at the center of the loading bar, thereby producing a pure bending moment in the specimen free from transverse shear stresses. Lead counter weights at the ends of the holders *A* and *B* were used to counterbalance the weight of the holders. Provision was made also to vary the lever arm *x* (Fig. 2) and the height of the fulcrum *G*. The latter adjustment allows the maintenance of a horizontal position for the lever.

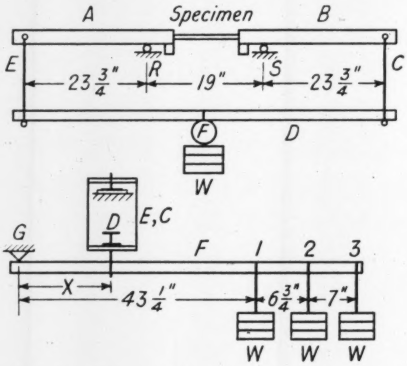


FIG. 2.—Dimensions of Test Apparatus.

Deformations were measured as shown in Fig. 4 by means of a deflection gage with 3-in. span and by strain gages of

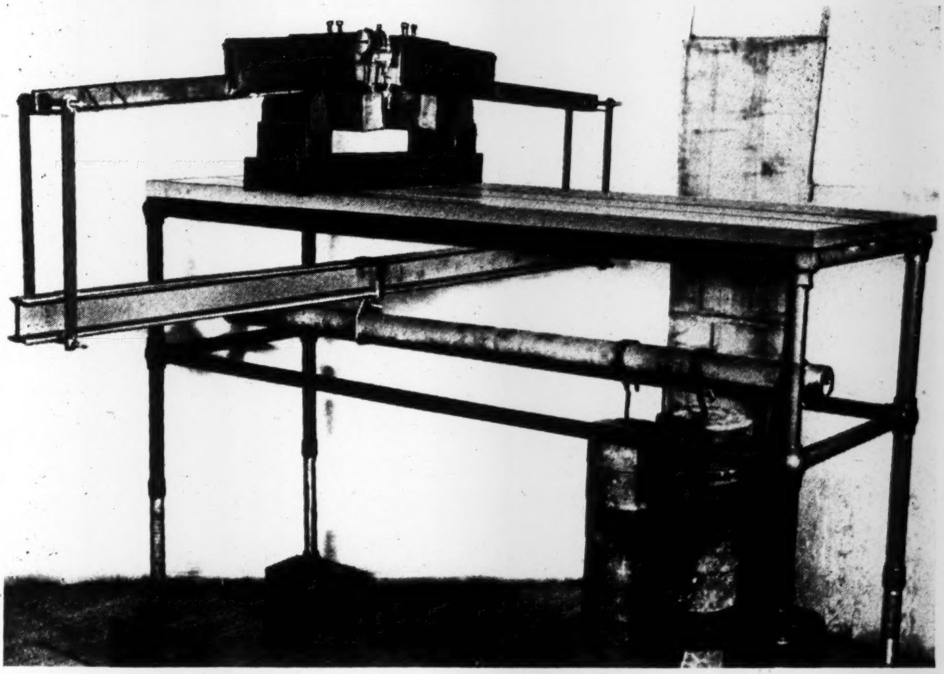


FIG. 3.—Test Apparatus for Plastic Bending Experiments.

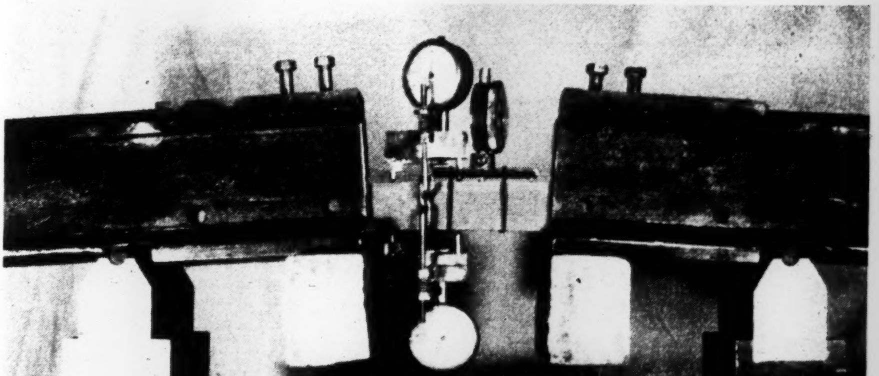


FIG. 4.—Gages for Measuring Strains and Deflections.

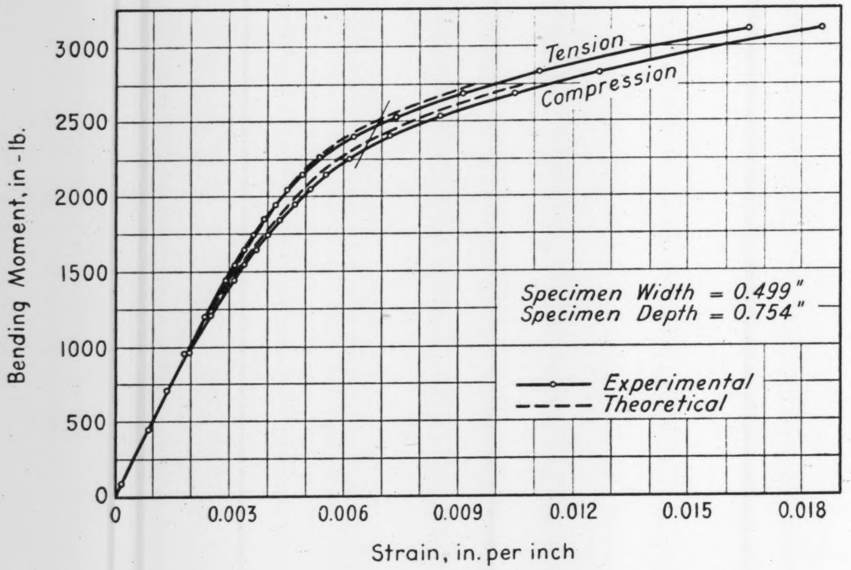


FIG. 5.—Relationship Between Strain and Bending Moment for Specimen No. 1.

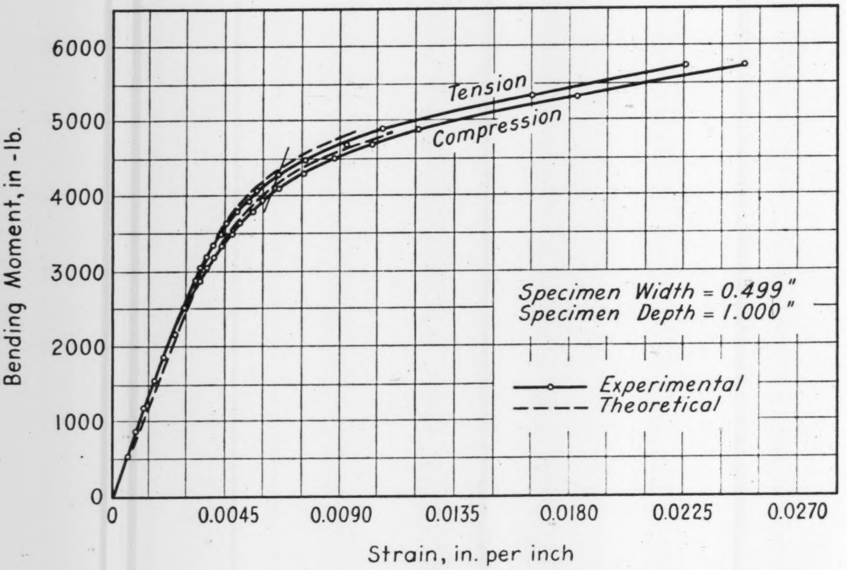


FIG. 6.—Relationship Between Strain and Bending Moment for Specimen No. 2.

the Berry type with a 2-in. gage length, having a two-to-one lever multiplication factor. All three gages had dials reading to 0.0001 in. The deflections and strains on the top and bottom fibers of the specimen could in this way be measured.

Test Procedure:

In the elastic range of loading, appropriate increments of load were

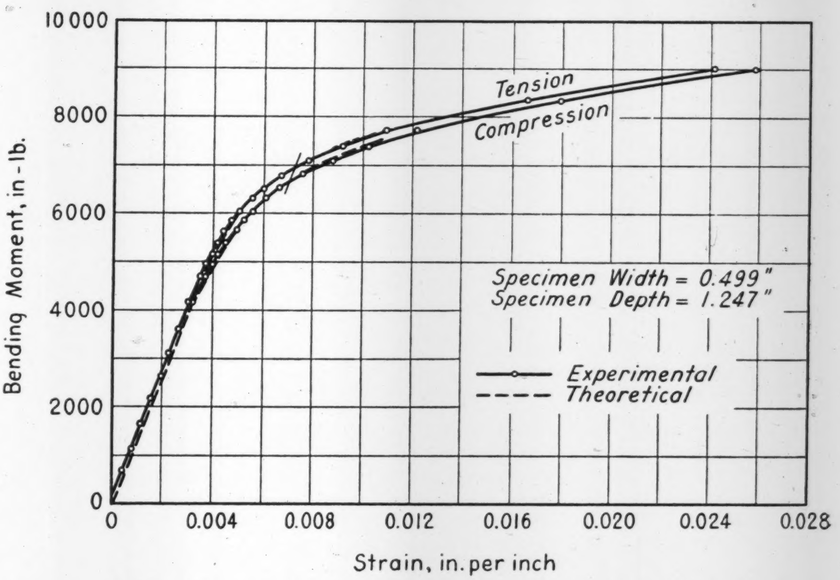


FIG. 7.—Relationship Between Strain and Bending Moment for Specimen No. 3.

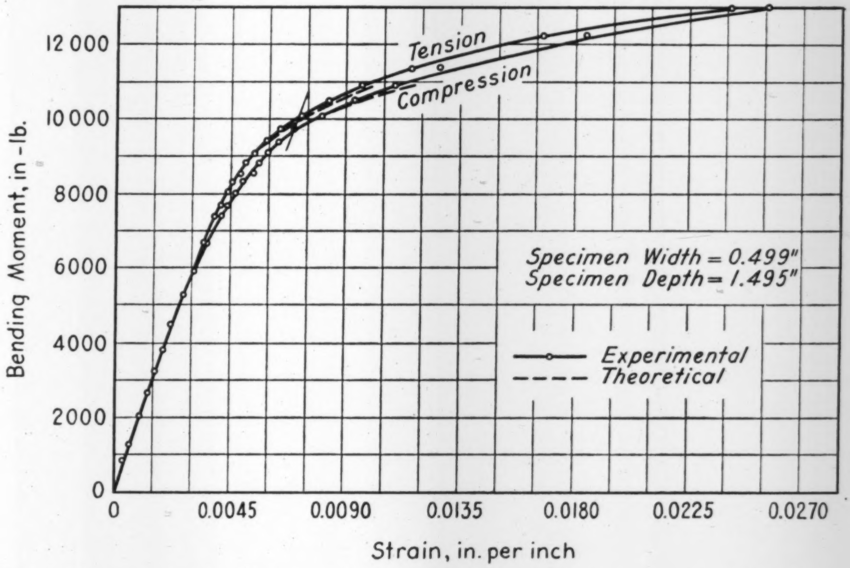


FIG. 8.—Relationship Between Strain and Bending Moment for Specimen No. 4.

applied and corresponding readings of the strains and deflections noted. In the plastic range beyond the yield load a difficulty was encountered due to the creep deformations which accompanied each application of load. An arbitrary time interval of 5 min. was used between the time the load was applied and the time the deformations were read. After this time interval a steady state was

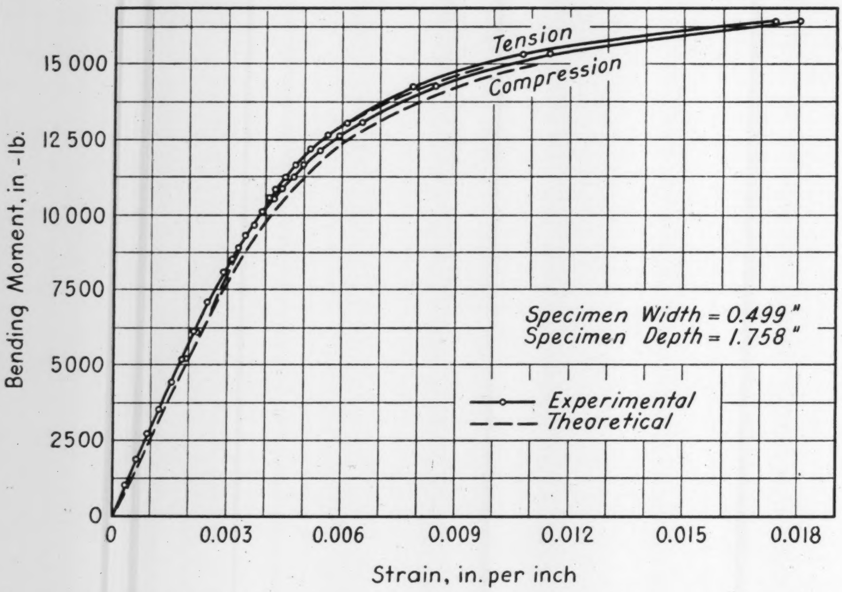


FIG. 9.—Relationship Between Strain and Bending Moment for Specimen No. 5.

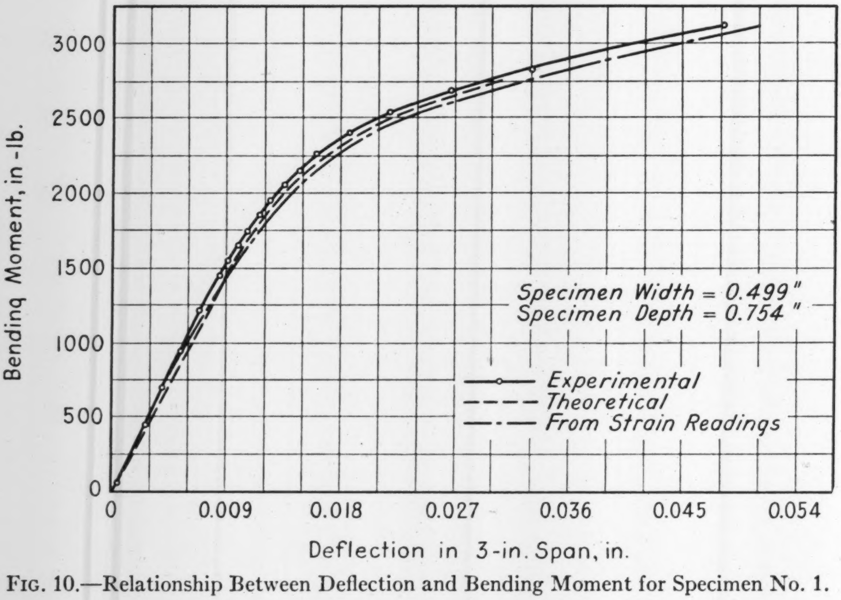


FIG. 10.—Relationship Between Deflection and Bending Moment for Specimen No. 1.

approximated. The influence of this time factor has been studied by several investigators, notably Nadai and Davis (9). The problem, however, needs further study.

It should be noted that this investigation is confined to the first part of the plastic range since, with excessive deflections, the interpretation of the strains and deflections would have to be corrected for curvature.

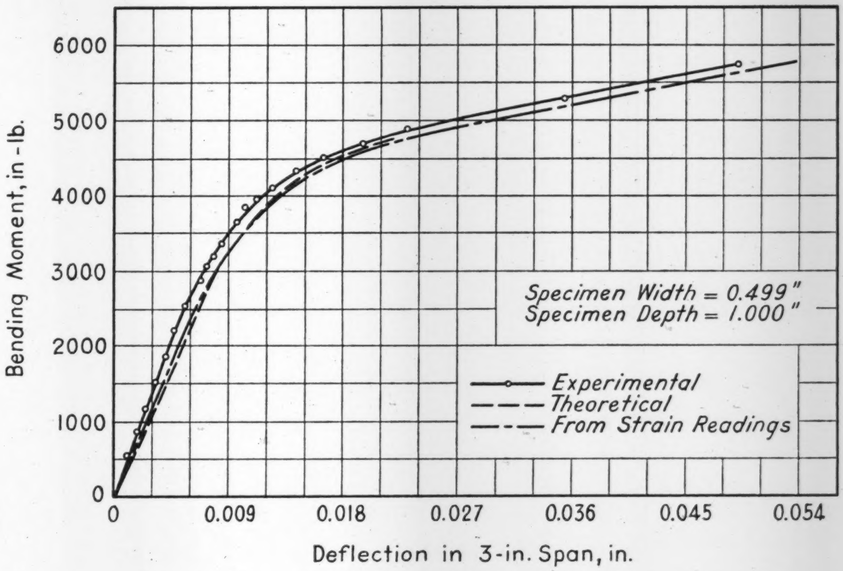


FIG. 11.—Relationship Between Deflection and Bending Moment for Specimen No. 2.

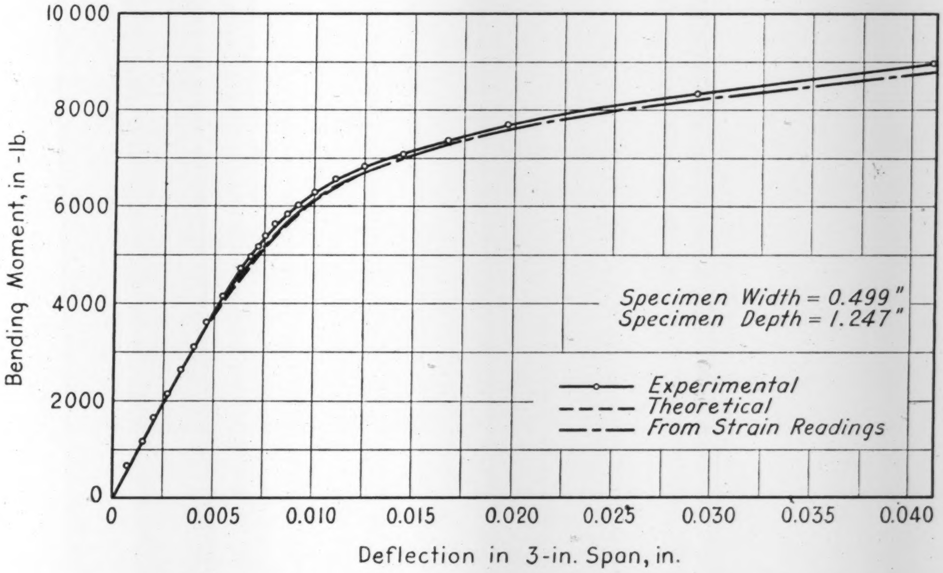


FIG. 12.—Relationship Between Deflection and Bending Moment for Specimen No. 3.

Test Results:

Relationships between the bending moment and the experimental values of the strains are shown in Figs. 5 to 9 for the five specimens tested. Similar curves for deflection are given in Figs. 10 to 14.

In order to interpret the foregoing results, stress-strain diagrams in pure tension and compression were obtained (Fig. 15). That is, a theory for deflec-

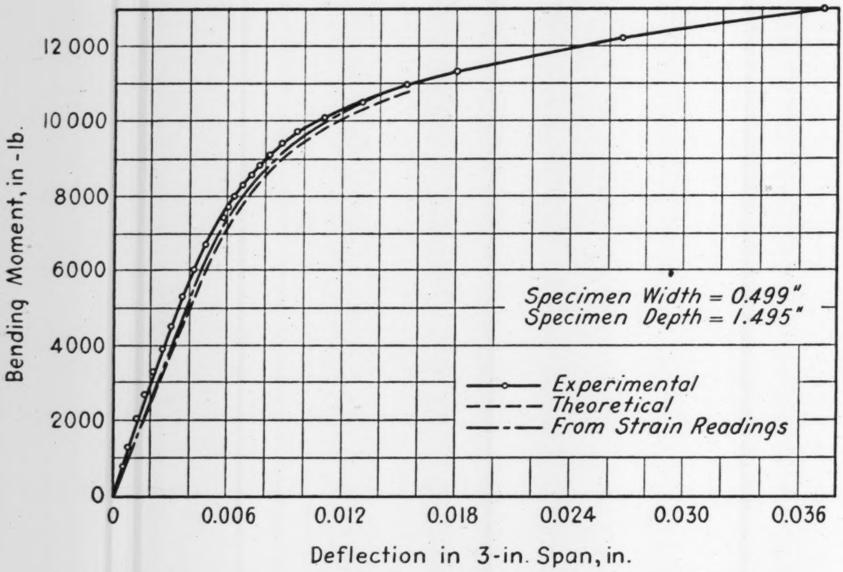


FIG. 13.—Relationship Between Deflection and Bending Moment for Specimen No. 4.

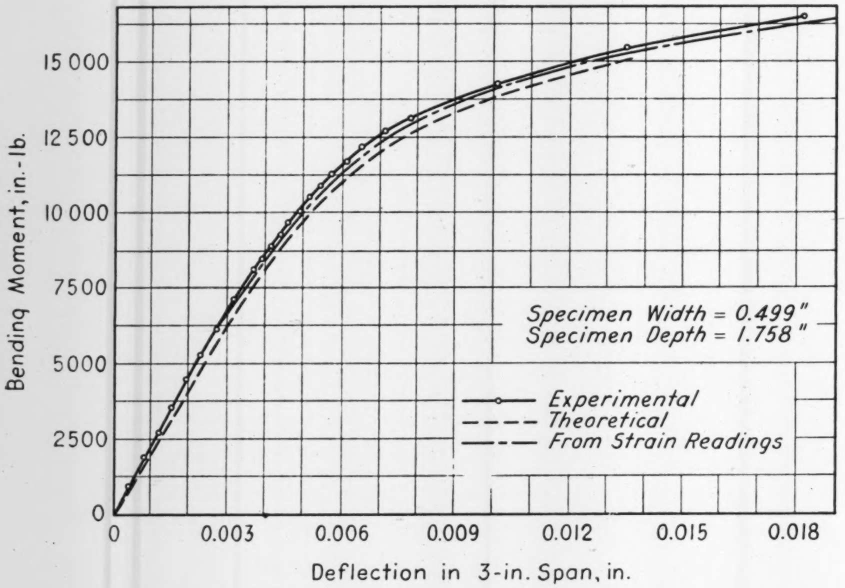


FIG. 14.—Relationship Between Deflection and Bending Moment for Specimen No. 5.

tions and strains in plastic bending can be developed based on the simple tension and compression stress-strain diagrams. A comparison of these theoretical results with the actual bending test data indicates the accuracy of this interpretation. The test results in Fig. 15 were supplied by R. L. Templin, Chief of Tests, Aluminum Company of America, New Kensington, Pa.

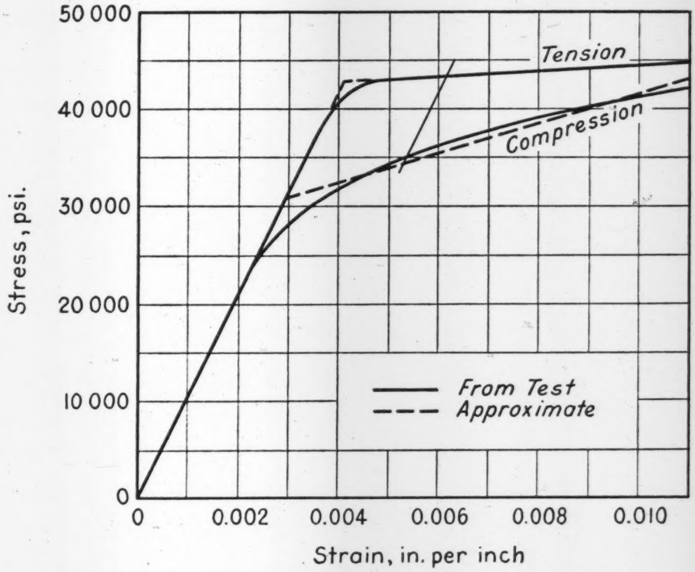


FIG. 15.—Stress-Strain Relationship for 17S-T Aluminum Alloy in Pure Tension and Compression.

INTERPRETATION OF TEST RESULTS

In interpreting the plastic bending test results, the object is to see whether it is possible approximately to predict behavior in plastic bending based on simple tension and compression test data. For this purpose an approximation to the tension and compression diagrams is made by replacing them with two straight lines as shown in Fig. 15. It should be noted that this may not be a satisfactory assumption for some materials nor for the latter part of the stress-strain diagram. The interpretation given herein, however, is restricted to the first part of the plastic range.

Theoretical Determination of Strains and Deflections in Plastic Bending:

Figure 16 represents a prismatic bar subjected to a pure bending moment between the reactions P. This is the loading condition used in the foregoing tests. Under this pure bending moment with no transverse shearing forces on the specimen, it can be shown by the theory of elasticity (10) that the only stresses present are horizontal tensile stresses above the neutral axis and horizontal compressive stresses below the neutral axis.

To determine the theoretical relations between the bending moment and strains on the outer fibers based on

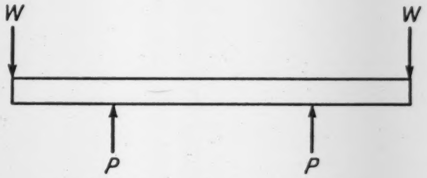


FIG. 16.—Beam in Pure Bending.

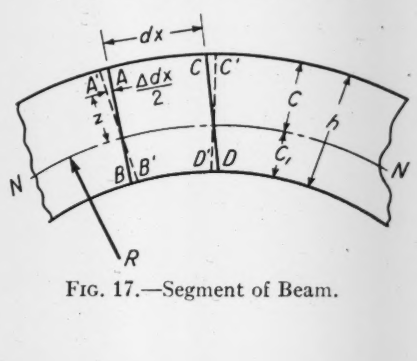


FIG. 17.—Segment of Beam.

simple tension and compression stress-strain diagrams, the general theory of plasticity as given by Nadai (1) will be used. In this theory the assumption is made that a plane cross-section of the member in bending remains plane after bending. That is, in Fig. 17 two sections AB and CD a distance dx apart rotate to $A'B'$ and $C'D'$, respectively. Then if (Δdx) is the extension of a fiber at a distance z from the neutral axis, the unit strain is

$$e = \frac{(\Delta dx)}{dx} \ldots\ldots\ldots\ldots\ldots (1)$$

From similar triangles,

$$\frac{(\Delta dx)}{dx} = \frac{z}{R} \ldots\ldots\ldots\ldots\ldots (2)$$

where $R =$ the radius of curvature of the neutral axis. Then from Eqs. 1 and 2,

$$e = \frac{z}{R} \ldots\ldots\ldots\ldots\ldots (3)$$

For the outer fibers, if e_y equals the unit strain on the tension side and $e_{y'}$ the unit strain on the compression side, then from Eq. 3,

$$e_y = \frac{c}{R} \quad \text{and} \quad e_y' = \frac{c_1}{R}$$

or

$$R = \frac{h}{e_y + e_y'} \ldots\ldots\ldots\ldots\ldots (4)$$

where $h =$ the depth of the beam.

For equilibrium, the sum of the stresses on the section must be zero and the sum of the moments of the stresses must equal the applied moment M. Then if S equals the unit stress on an area dA at a distance z from the neutral axis, these equilibrium conditions can be written

$$\int_{-c_1}^{c} S\,dA = 0 \ldots\ldots\ldots\ldots (5)$$

and

$$\int_{-c_1}^{c} Sz\,dA = M \ldots\ldots\ldots\ldots (6)$$

For a rectangular cross-section $dA = bdz = bRde$, and Eqs. 5 and 6 become

$$\int_0^{e_y} S\,de = \int_{-e_y'}^{0} S\,de \ldots\ldots\ldots\ldots (7)$$

and

$$M = bR^2 \int_{-e_y'}^{e_y} Se\,de$$

or

$$M = \frac{bh^2}{(e_y + e_y')^2}\left[\int_0^{e_y} Se\,de + \int_{-e_y'}^{0} Se\,de\right] \ldots (8)$$

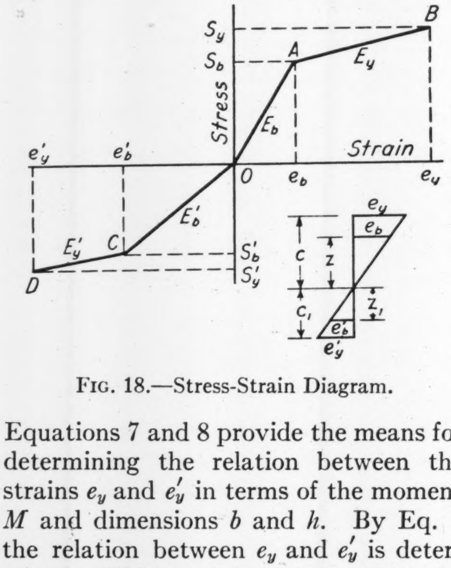


FIG. 18.—Stress-Strain Diagram.

Equations 7 and 8 provide the means for determining the relation between the strains e_y and e_y' in terms of the moment M and dimensions b and h. By Eq. 7 the relation between e_y and e_y' is determined. This equation states that the areas under the stress-strain diagrams in tension and compression are equal.

The determination of the moment-strain relations can be obtained graphically by integrating the areas under the stress-strain diagrams. This was done in this investigation but it was found that the results differed only slightly from those obtained assuming the simple tension and compression diagrams as

TABLE I.—THEORETICAL STRAIN-MOMENT AND DEFLECTION VALUES.

Unit Strain, e_y, in. per inch	Unit Strain, e'_y, in. per inch	Factor, k	Moments and Deflections for each Strain, e_y									
			h = 0.754 in.		h = 1.000 in.		h = 1.247 in.		h = 1.495 in.		h = 1.758 in.	
			M	δ	M	δ	M	δ	M	δ	M	δ
0.001	0.001[a]	10 300	487	0.0030	856	0.0023	1330	0.0018	1920	0.0015	2640	0.0013
0.002	0.002[a]	20 600	973	0.0060	1712	0.0045	2660	0.0037	3840	0.0030	5290	0.0026
0.003	0.003[a]	30 900	1460	0.0090	2570	0.0068	3990	0.0054	5760	0.0045	7930	0.0038
0.0035	0.00352[b]	35 600	1680	0.0105	2960	0.0079	4600	0.0064	6630	0.0053	9150	0.0045
0.004	0.00413[b]	40 100	1892	0.0121	3330	0.0091	5180	0.0073	7470	0.0061	10 300	0.0052
0.005	0.0054	46 300	2190	0.0161	3770	0.0117	5980	0.0094	8630	0.0079	11 900	0.0067
0.006	0.0066	50 100	2370	0.0188	4170	0.0142	6480	0.0114	9330	0.0095	12 870	0.0081
0.007	0.0078	52 800	2490	0.0221	4390	0.0167	6820	0.0134	9840	0.0112	13 570	0.0095
0.008	0.0089	55 200	2610	0.0252	4580	0.0190	7130	0.0153	10 300	0.0128	14 180	0.0108
0.009	0.0100	56 900	2690	0.0284	4730	0.0214	7535	0.0172	10 600	0.0143	14 600	0.0122
0.010	0.0110	58 400	2760	0.0324	4860	0.0237	7550	0.0190	10 880	0.0158	15 000	0.0134

[a] Both elastic.
[b] One elastic, one plastic.

two straight lines (Fig. 15). For this reason Eqs. 7 and 8 will be applied to the stress-strain diagram of Fig. 15, as shown diagrammatically in Fig. 18. In the Appendix the interpretation of tests is given using parabolic stress-strain relations.[4]

In Fig. 18, S_y corresponds to the stress on the outer fiber in tension and S'_y to the stress on the compression side. E_b, E_y, E'_b, and E'_y are the slopes of the lines OA, AB, OC, and DC, respectively. Referring to Fig. 18,

$$e_y = e_b + \frac{S_y - S_b}{E_y}$$

or

$$\left.\begin{aligned} S_y &= E_y e_y - e_b(E_y - E_b) \\ &\text{Similarly,} \\ S'_y &= E'_y e'_y - e'_b(E'_y - E'_b) \end{aligned}\right\} \dots\dots (9)$$

Using Eq. 8, the summation of the moments of these stresses is

$$\begin{aligned} M = \frac{b}{6}[(c^2 + cz)S_b &+ (c_1^2 + c_1 z_1)S'_b \\ &+ (2c^2 - cz - z^2)S_y \\ &+ (2c_1^2 - c_1 z - z_1^2)S'_y] \,..\,(10) \end{aligned}$$

Since plane sections are assumed to remain plane,

$$z = c\,\frac{e_b}{e_y}, \qquad z_1 = \frac{ce'_b}{e_y}, \qquad c_1 = c\,\frac{e'_y}{e_y}$$

$$\text{and} \quad c = \frac{he_y}{(e'_y + e_y)} \dots\dots\dots (11)$$

Placing values of z, z_1, and c_1 from Eq. 11 in Eq. 10,

$$\begin{aligned} M = \left(\frac{bh^2}{6}\right)\left(\frac{1}{e'_y + e_y}\right)&[e_y(e_y + e_b)E_b e_b \\ &+ e'_y(e'_y + e'_b)E'_b e'_b \\ &+ (2e_y^2 - e_b e_y - e_b^2)(E_y e_y - E_y e_b + E_b e_b) \\ &+ \{2(e'_y)^2 - e'_y e'_b - (e'_b)^2\} \\ &\quad \times \{E'_b e'_y - E'_y e'_b + E'_b e'_b\}] \,..\,(12) \end{aligned}$$

$$\text{or} \quad M = k\left(\frac{bh^2}{6}\right) \dots\dots\dots (12)$$

Using Eq. 7, and for the stress-strain diagram assumed,

$$S_b \cdot \frac{bz}{2} + (S_b + S_y)(c - z)\frac{b}{2}$$

$$= \frac{S'_b b z_1}{2} + \left(\frac{S'_b + S'_y}{2}\right)(c_1 - z_1)b \,..\,(13)$$

---

[4] See p. 595.

Substituting values of z, z_1, and c_1 from Eq. 11 in Eq. 13 and solving for e_y',

$$e_y' = -e_b'\left(\frac{E_b'}{E_y'} - 1\right) \pm \sqrt{(e_b')^2\left[\left(\frac{E_b'}{E_y'}\right)^2 - \frac{E_b'}{E_y'}\right] + \frac{E_b}{E_y'}(2e_b e_y - e_b^2) + \frac{E_y}{E_y'}(e_y - e_b)^2} \quad \text{. . (14)}$$

By using Eqs. 12 and 14 the relation between the moment M and strains e_y and e_y' can be determined. The procedure for doing this is as follows:

1. From the stress-strain diagram in simple tension and compression find values of e_b, e_b', E_b', E_b, E_y, and E_y'.
2. Using Eq. 14 determine e_y' for different values of e_y.
3. Using Eq. 12 values of M can be determined for the values of e_y selected.

The relation between the curvature R and moment M can now be determined by Eq. 4. The maximum deflection δ for a gage length L, in terms of the radius R is, approximately,

$$\delta = \frac{L^2}{8R} \quad \text{. . . . . (15)}$$

Combining Eqs. 5 and 15, the deflection δ in terms of the strains e_y and e_y' becomes

$$\delta = \frac{L^2(e_y + e_y')}{8h} \quad \text{. . . . . (16)}$$

Using the values of e_y and e_y' in terms of M as obtained from Eqs. 14 and 12, Eq. 16 gives the theoretical moment-deflection relationship.

In applying the foregoing equations to a particular calculation it will be necessary to observe three possible stages of loading and to modify Eqs. 14 and 12 for each case. These conditions are:

Case 1—General Case.—The fibers in both tension and compression are stressed elastically on the center fibers and plastically on the outer fibers. Equations 12 and 14 apply as given.

Case 2—Special Case.—Both sides of the beam are in the elastic range. Equations 12 and 14 can be used by placing $E_y = E_b$ and $E_y' = E_b'$. This reduces to the common beam formula.

Case 3—Special Case.—One side of the beam is entirely in the elastic range while the other is partly elastic and partly plastic. Equations 12 and 14 are modified by placing $E_y = E_b$ or $E_y' = E_b'$.

The values of the theoretical moment M corresponding to various magnitudes of e_y are given in Table I for beams with dimensions equal to those tested. The moments were calculated using Eqs. 12 and 14. Table I also shows the theoretical deflection values based on Eq. 16 for various values of the moments.

Comparison of Experimental and Theoretical Values of Strains and Deflections:

The moment and strain values in Table I are plotted in Figs. 4 to 9 for purpose of comparison with test results. The deflection and moment values are similarly plotted in Figs. 9 to 14. An inspection of these graphs shows a very good agreement between test results in plastic bending and theoretical values based on simple tension-compression relations.

Determination of Yield Strength in Bending:

The determination of the yield strength values of the beams tested will be determined on the basis of an equivalent offset strain method (11). This method determines an offset deflection for finding the yield strength in bending which is equivalent to the corresponding A.S.T.M. offset strain used for defining yield strength in tension. It correlates yield strength in bending and tension and appears, to the authors, as preferable to the usual Johnson's apparent elastic limit method used for finding yield strength in bending.

To explain the equivalent offset deflection method, consider the yield strength in tension as defined by point A in Fig. 19 (a). This point corresponds to a unit strain e_f and the problem is to find a deflection δ_f in the moment-deflection diagram of Fig. 19 (b) which is equivalent to this strain. From Fig. 19 (a),

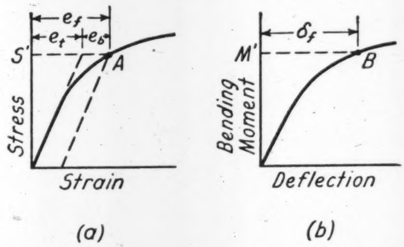


FIG. 19.—Correlation of Yield Points in Tension and Bending.

$$e_f = e_t + e_b = \frac{S'}{E} + e_b \text{.......} (17)$$

By Eq. 16, if $e'_y = e_y = e_b$,

$$\delta_f = \frac{e_b L^2}{4h} \text{............} (18)$$

The strains e_b and e_f as given by Eqs. 17 and 18 are now made equal in order to determine the equivalent offset deflection. Then

$$\delta_f = e_f \frac{L^2}{4h} = \left(\frac{S'}{E} + e_b\right) \frac{L^2}{4h} \text{....} (19)$$

Since all the quantities on the right-hand side of Eq. 19 are known, the offset deflection δ_f can be determined for each beam tested. Then the values of the bending moment at yield, M', are obtained from Figs. 10 to 14. Assuming that Hooke's law applies to this yield point, the yield strength in bending is

$$S'_b = \frac{M'h}{2I} \text{............} (20)$$

The yield stress values for each beam, calculated by Eq. 20, are given in Table II. The ratio of these yield stresses in bending to the yield stresses in tension are given in the last column of Table II. These ratios are in remarkably close agreement but there are not sufficient data to arrive at a definite conclusion.

CONCLUSIONS

This study shows how tension and compression stress-strain diagrams can be used to determine strain-moment and deflection-moment relationships in plastic bending. Within the limits of the test results given, a comparison of the theoretical results with experimental values shows a good agreement. The yield strength in bending, as determined by an equivalent offset method, gives consistent values for different ratios of width to depth of beam. There are not sufficient experimental results given, however, to warrant a definite conclusion on this result.

TABLE II.—YIELD STRENGTH IN BENDING.

Depth of Beam, in.	Ratio of Depth to Width	Section Modulus, in.^{3}	Deflection at Yield, in.	Moment at Yield, in-lb.	Yield Strength, psi.	Strength Ratio, Bending to Tension
0.754	1.51	0.047	0.0186	2 400	50 800	1.17
1.000	2.00	0.083	0.0141	4 300	51 700	1.19
1.247	2.50	0.129	0.0113	6 620	51 200	1.18
1.495	3.00	0.186	0.0094	9 700	52 100	1.12
1.758	3.52	0.257	0.0080	13 200	51 300	1.18

Acknowledgments:

The authors acknowledge with thanks the help of P. C. Huntly of Illinois Institute of Technology for his encouragement, and R. L. Templin of the Aluminum Company of America for the contribution of the material tested. This paper was prepared in part from a thesis of F. D. Cotterman prepared under the direction of Joseph Marin.

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APPENDIX

INTERPRETATION OF TESTS USING PARABOLIC STRESS-STRAIN RELATION

For some materials the stress-strain relation in simple tension and compression can be represented by the equation of a parabola. That is, for tension and compression the stress-strain relations can be assumed, respectively, as

$$E = \frac{S_y^n}{e} \quad \text{and} \quad E' = \frac{(S_y')^m}{e'} \ldots\ldots \text{(a)}$$

where E, n, E', and m are experimental constants.

For equilibrium, the moment M for a rectangular section is

$$M = \int_0^c S_y z b\, dz + \int_0^{c_1} S_y' z_1 b\, dz_1 \ldots \text{(b)}$$

From the condition of plane sections, and using Eq. (a), the stresses S_y and S_y', in terms of the stresses S and S' on the outer fiber, are

$$S_y = S\left(\frac{z}{c}\right)^{\frac{1}{n}} \quad \text{and} \quad S_y' = (S')\left(\frac{z_1}{c_1}\right)^{\frac{1}{m}} \ldots \text{(c)}$$

Placing values of S_y and S_y' from Eq. (c) in Eq. (b),

$$M = \frac{Sn(bc^2)}{(2n+1)} + \frac{S'm(bc_1^2)}{(2m+1)} \ldots\ldots \text{(d)}$$

From Eqs. (a) and (c),

$$S' = S^{\frac{n}{m}}\left[\frac{c_1 E'}{cE}\right]^{\frac{1}{m}} \ldots\ldots\ldots\ldots \text{(e)}$$

To determine the distances c_1 and c from the neutral axis to the outer fibers, the condition that the summation of the bending stresses must equal zero is used. That is,

$$\int_0^c S_y b\, dz = \int_0^{c_1} S_y' b\, dz_1 \ldots\ldots\ldots \text{(f)}$$

Placing values of S_y and S_y' from Eqs. (c) in Eq. (f),

$$c_1 = \left(\frac{1+m}{1+n}\right)\left(\frac{n}{m}\right)\left(\frac{S}{S'}\right)c \ldots\ldots \text{(g)}$$

Since $c_1 + c = h$, the values of c_1 and c are

$$\left.\begin{aligned} c &= \frac{h(1+n)mS'}{(1+n)mS' + (1+m)nS} \\ \text{and} \\ c_1 &= \frac{h(1+m)nS}{(1+n)mS' + (1+m)nS} \end{aligned}\right\} \ldots \text{(h)}$$

Placing the value of S' from Eq. (e) in Eq. (h) and, in turn, the values of c and c_1 from Eq. (h) in Eq. (d),

$$M = \frac{b}{[(1+n)mS' + (1+m)nS]_2} \times \left[h^2(1+n)^2 m^2 (S')^2 \frac{Sn}{2n+1} + \left\{\frac{E'(1+m)nS}{E(1+n)mS'}\right\}^{\frac{1}{m}} \{h(1+m)nS\}^2 \times \frac{(S^{\frac{n}{m}} m)}{2m+1}\right] \ldots\ldots\ldots\ldots\ldots\ldots \text{(i)}$$

By means of Eqs. (e) and (i) the stress S or S' is completely defined in terms of the moment M, dimensions b and h, and material constants E, E', n, and m. Knowing the stresses on the outer fibers in terms of the moment, the corresponding strains can be calculated by Eq. 12. With these strains known the deflection-moment relation can be found by Eq. 13.

DISCUSSION

MR. DON S. WOLFORD[1] (*presented in written form*).—The close agreement of the authors' experimental and theoretical data can only be the result of carefully conducted tests correlated with sound mathematical analysis. They have wisely selected the rectangular section for their proof. The rectangular section not only lends itself to relatively easy mathematical treatment, but by being one of the most commonly used, merits positive verification before more complicated sections are studied. Furthermore, 17S-T aluminum alloy is an appropriate material for such a correlation.

The recent emphasis on compression testing of aircraft materials reveals that some, like the 17S-T aluminum alloy used by the authors, have unequal compressive and tensile properties. Such inequalities are also observed in ferrous alloys, as for example, the cold-worked austenitic stainless steels. The excellent agreement obtained by Messrs. Marin and Cotterman should encourage other investigators in developing better design correlations based on material properties.

The conventional approach to the solution of the problem of plastic bending has been by means of the concept of the reduced modulus of elasticity in bending, summarized by Timoshenko.[2] The advantage in using the reduced modulus is that the material properties and section factors can be lumped in the one mathematical operation, simplifying the subsequent calculations of strength and deflection. We applied this method to the authors' data and found it in good agreement. The authors mention having tried a similar method and found "the results differed only slightly from those obtained assuming the simple tension and compression diagrams as two straight lines." The reduced modulus of a solid rectangular section is proportional to the moment-area bounded by the stress-strain curve about the stress-axis, and can be approximated by means of the trapezoidal rule of integration. In our check, we determined separate reduced moduli for tension and compression because these were unequal, since the inequality indicates a shift in the location of the neutral axis, making necessary the separate treatment of tension and compression parts of the beam. The shift of the neutral axis as the beam was bent into the plastic range was located from the areas of the tensile and compressive stress-strain diagrams.

In a recent article, Cozzone[3] arrives at bending strength in the plastic range by assuming an equivalent trapezoidal stress distribution. His method consists of determining an "intercept stress" for a given material from its stress-strain curve and subsequently applying it in his formula for bending strength. This formula contains a term called the "section factor," which compensates for the section's shape. Cozzone indicated he thought the differences between tensile and compressive properties were minor compared to his other factors, and consequently neglected them. By the application of the reduced modulus to Messrs. Marin and Cotterman's data, it can be shown that the shift in the neutral axis cannot be ignored in close correlations. The reduced modulus in bending for either tension or compression can be obtained from the following expression:

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[1] Research Engineer, The American Rolling Mill Co., Middletown, Ohio.

[2] S. Timoshenko, "Theory of Elastic Stability," First Edition, pp. 45–63, McGraw-Hill Book Co. Inc., New York, N. Y.

[3] Frank P. Cozzone, "Bending Strength in the Plastic Range," *Journal of the Aeronautical Sciences*, Vol. 10, No. 5, May, 1943, pp. 137-151.

$$E_r = \frac{3}{e^3}\int_0^e Sede \ldots\ldots (1)$$

where:

E_r = reduced modulus of elasticity in pounds per square inch,

e = strain in inches per inch, and

S = stress in pounds per square inch.

The use of the above formula requires a function of stress in terms of strain which ordinarily is difficult to determine and even harder to apply. We divided the stress-strain curves into vertical strips each having a width along the strain axis of 0.0005 in. per inch. The following expression gives the entire moment-area up to the proportional limit:

$$\text{moment-area} = \frac{S_p e_p^2}{3} \ldots\ldots (2)$$

where the subscript "p" indicates the station nearest the proportional limit.

After the proportional limit is exceeded, moment-areas of individual strips can be approximated as follows:

$$\text{moment-area} = \frac{(S_x + S_{(x+1)})(e^2_{(x+1)} - e^2_x)}{4} \ldots (3)$$

where "x" indicates any strip station.

The reduced modulus is then $\frac{3}{e_x^3} \times$ (total moment-area to station "x"). The curves for reduced section are given in Fig. 1.

Let $K = y'/y$, where K becomes the ratio of the maximum fiber distances on the compression and tension sides of the rectangular section.

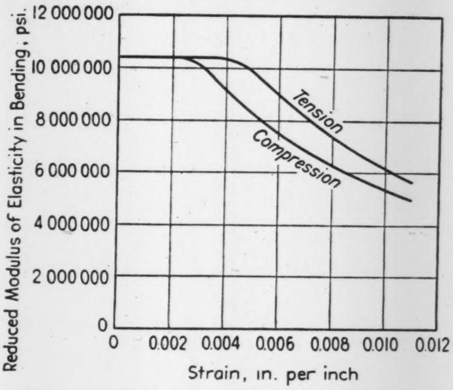

FIG. 1.—Relation of Reduced Modulus of Elasticity in Bending to Strain for 17S-T Aluminum Alloy (Based on Fig. 15 of Paper).

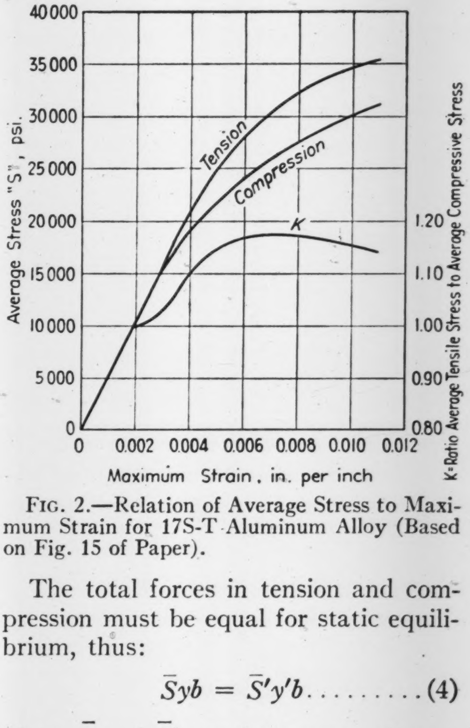

FIG. 2.—Relation of Average Stress to Maximum Strain for 17S-T Aluminum Alloy (Based on Fig. 15 of Paper).

The total forces in tension and compression must be equal for static equilibrium, thus:

$$\bar{S}yb = \bar{S}'y'b \ldots\ldots\ldots (4)$$

where $\bar{S}$ and $\bar{S}'$ are the average stresses up to the maximum stresses S_y and S_y' at extreme fiber distances y and y', respectively, and b is the width of the section.

By substitution

$$K = \bar{S}/\bar{S}' \ldots\ldots\ldots\ldots (5)$$

The average stresses were obtained by the trapezoidal approximation to the following exact expression

$$\text{Average stress} = \frac{1}{e}\int^{e} Sde \quad \ldots (6)$$

The average stresses and K are given in the accompanying Fig. 2 in relation to the maximum strain.

The tension and compression parts of the rectangular section may be considered separately. The bending moment would then be:

$$M = \frac{E_r I}{\rho} + \frac{E'_r I'}{\rho} \quad \ldots\ldots (7)$$

since $K = y'/y$
$e' = K e$
$e = y/\rho$ and $e' = y'/\rho$
$h = y' + y$

By substitution

$$M = \frac{bh^2 e}{3(K + 1)^2}\,(E'_r K^3 + E_r) \quad \ldots (8)$$

where E_r and E'_r are the reduced moduli in tension and compression, respectively, and e is the corresponding tensile strain. The above expression has been applied to specimen No. 3 with the results given in the accompanying Fig. 3, and shows close agreement between the experimental values obtained by the authors and the theoretical curves calculated from the reduced modulus. These computations showed that in the plastic range, the weaker compressive part of the beam actually contributed 20 per cent more resisting moment at the yield strength than the stronger tension part. The appearance of the factor K in the third degree (Eq. 8) accounts for this proportion of the moments.

Solid rectangular sections are but one of many used in structural design. Hollow sections with as much material remote from the neutral axis as practical are more common in aircraft. The derivation of accurate design formulas for sections other than rectangular has been impeded by the complexity of the mathematical treatments involved. The reduced modulus in bending for a section with its material ideally remote from the neutral axis is the secant modulus[5] which can be determined directly from the stress-strain curve.[4] The reduced moduli for solid rectangular and ideally remote sections probably represent the extremes bracketing most sections. The designer should be able to estimate the probable deflection of any stable section by applying these reduced moduli representing the extremes to the indeterminent section.

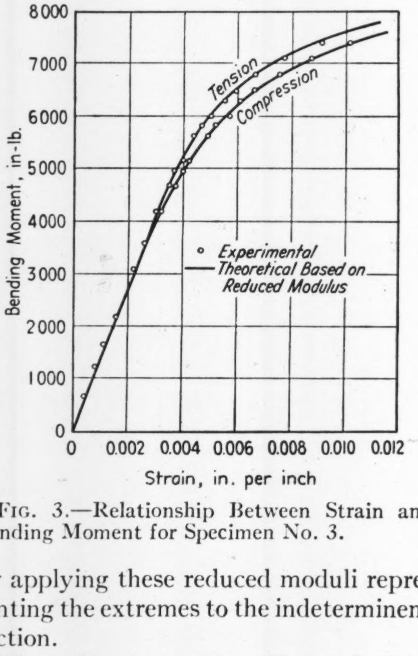

FIG. 3.—Relationship Between Strain and Bending Moment for Specimen No. 3.

MR. ROBERT H. HEYER.[5]—The authors' tests were made on deep sections using a gradually yielding material. The yield strength ratios—bending to tension—average 1.17, or 17 per cent higher in bending.

We have had occasion to make a large number of bending tests on small sheet samples using concentrated center loading.

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[4] D. S. Wolford, "Significance of the Secant and Tangent Moduli of Elasticity in Structural Design," *Journal of the Aeronautical Sciences*, Vol. 10, No. 6, June, 1943, pp. 169–179.

[5] Supervisor, Metallurgical Service Laboratories, The American Rolling Mill Co., Middletown, Ohio.

In the case of sharp-yielding annealed low-carbon steels, we have always obtained a much higher ratio of bending to tensile yield point. For example, in one group of 360 tests, this ratio was 1.59, or 59 per cent higher in bending than in tension.

This is in approximate agreement with the theoretical factor of 1.50 which Cozzone[3] has recently given for rectangular sections and 1.46 which Kennedy determined for 0.29 per cent carbon steel beams of various cross-sections.

MR. A. NADAI.[6]—In this paper, the authors are using the power function law to express stress-strain diagrams for tension or compression. In some cases of their applications they also use the stress-strain diagram of Fig. 18 which consists of four straight lines.

I should like to make a suggestion, which could be of assistance for solving similar problems.

Referring in particular to stress-strain curves of the shape shown in the authors' Fig. 19 (*a*) which show a gradual deviation from the straight line corresponding to the elastic strains, the suggestion which I like to make is to introduce two additive portions for the strains, namely, an elastic portion ϵ' and a permanent portion ϵ''. Thus for the total strain ϵ is assumed:

$$\epsilon = \epsilon' + \epsilon'' \ldots\ldots\ldots (9)$$

For the elastic portion we write $\epsilon' = \sigma/E$ and for the plastic portion we may use $\epsilon'' = f(\sigma)$ in general or the same expression as that given by the authors in their Eq. (a) in the appendix to the paper, namely

$$\epsilon'' = c\sigma^n \ldots\ldots\ldots\ldots (10)$$

(σ = stress, E = modulus of elasticity, c and n = constants) provided that it fits the experimentally found shape of the stress-strain curve. The stress-strain curve appears in the form finally:

$$\epsilon = \frac{\sigma}{E} + c(\pm\sigma)^n \ldots\ldots (11)$$

c and n may be different constants for the tension or for the compression portion of the stress-strain curve.

Expressions of this latter kind were tried with good results by Holmquist and the writer for determining the plastic collapse pressures of heavy-walled steel casings under external pressure.[7]

MESSRS. JOSEPH MARIN[8] AND F. D. COTTERMAN[9] (*authors' closure, by letter*).—Mr. R. H. Heyer refers to the ratio of yield strength in bending to yield strength in tension. This ratio is not necessarily the same for various ductile materials and even for the same material the ratio will depend somewhat on the method used in defining the yield strength in tension and bending.

The suggestion of Mr. Nadai to use a stress-strain relation as given by Eq. 11 of his discussion is an excellent one. His proposal permits the analysis of stress-strain curves which are initially straight, while the curves referred to in the Appendix contain no straight portion. There was not sufficient time, however, to try this proposal.

Mr. D. S. Wolford's verification of the interpretation of the test results using the reduced modulus of elasticity is appreciated. The application of the procedure used by Cozzone to the test data is also of interest. There are many types of cross-sections and materials to be considered before any conclusions can be made regarding the correlation between the tensile and compressive stress-strain relations and the bending stress-strain relation.

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[6] Consulting Engineer, Westinghouse Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[7] T. J. Holmquist and A. Nadai, "A Theoretical and Experimental Approach to the Problem of Collapse of Deep-Well Casing," *Journal*, Am. Petroleum Inst., p. 392 (1939).

[8] Professor of Engineering Mechanics, The Pennsylvania State College, State College, Pa.

[9] Research Engineer, Crane Co., Chicago, Ill.

THE TESTING AND PROPERTIES OF LOW-TIN SOLDERS LISTED IN RECENT FEDERAL SPECIFICATIONS

By J. A. Kies[1] and W. F. Roeser[1]

Synopsis

Since the recent War Production Board order limiting the tin content of solders for certain purposes to 20 per cent, there is need for information concerning the utility of solders in this class. The choice of a single basis for comparison of these solders with one another and with those popular in pre-war days is difficult, especially with the current lack of a standardized test procedure for soldered joints. The solders investigated were a commercial 50-50 tin-lead solder, four solders corresponding in composition to those designated by B, D, E, and G in Federal Specification QQ-S-571a, and a solder corresponding to M in Federal Specification E-QQ-S-571a. The principal variables affecting the results of strength tests of soldered joints are discussed. Results of short-time tests of single lap joints in sheet and strip specimens showed that there was little difference between the strengths achieved with the six solders investigated. More highly idealized tests would be expected to show greater differences between the solders. Considerable differences between the results obtained with different sheet materials were found. Some general agreement on a practical basis for comparing solders would be helpful at this time.

Early in 1943 the War Production Board issued a new General Preference Order M-43 designed to cut tin use in 1943 to about one half the 1941 consumption, a large portion of the saving to be effected by limiting the tin content of general-purpose solders to 20 per cent and restricting the uses to which tin-containing solders may be put. The "workability" or ease of application of low-tin solders and the strengths of joints obtainable with them are consequently of current interest.

In studying the literature on tests of soldered joints, one might easily become confused as to the relative merits of various solders because of the notable lack of uniformity of the reported results for a given solder. This is not a reflection on the validity of any published data but is a natural consequence of the many variables in soldering and testing not clearly specified when the results are given. It cannot be assumed that the highest strength reported is the only correct one and that all others are in error because of faulty soldering technique or some other reason. The following discussion of the principal variables will serve to indicate some of the difficulties met in attempts to compare various reported results. Data on some low-tin solders of particular interest at this time are also given.

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[1] Associate Metallurgist, and Principal Metallurgist, respectively, National Bureau of Standards, Washington, D. C.

FACTORS INFLUENCING THE RESULTS OBTAINED IN TESTING SOLDERED JOINTS

Types of Joints:

Most of the joints commonly used in the testing of solders may be classified as: the sleeve joint, the butt joint, the simple lap joint, or the double lap joint. For various reasons the results of strength tests on these joints should not always be expected to agree, especially when expressed as load per unit area. The results given in Table I illustrate the differences obtained with different types of joints.

TABLE I.—REPORTED STRENGTH OF SOLDERED JOINTS OF DIFFERENT TYPES—50-50 SOLDER.

Metals joined	Short-Time Shear Tests at Room Temperature, psi.				
	Rod and Sleeve Joint[a]	Ring and Plug Joint[b]	Butt Joint, Rods, [c]	Single Lap Joint, 0.25 in. long	Double Lap Joint, 0.25 in. long
Copper	5500	6100	7580	3300[d]	...
Steel	...	5600	...	4790[d] 8100[e]	...
Brass	...	5150	...	4200[d]	5300[f]

[a] Maupin and Swanger, reference (**2**).
[b] Nightingale and Hudson, reference (**1**).
[c] Rhines and Anderson, reference (**4**).
[d] Present investigation, fillet removed.
[e] Dowdell, Fine, Elliot, and Mattson, reference (**7**), fillets left on the work.
[f] Present investigation, fillets left on the work.

The sleeve joint and the double lap joint have the advantage of minimizing eccentricity of loading, that is, tensile components and bending can easily be made negligible in such joints during testing (**1**,**2**,**3**).[2] For this reason the strength of a sleeve or a double lap joint tends to exceed that of a simple lap joint when the results are expressed in terms of shear stress.

The butt type of joint was used by Rhines and Anderson (**4**) for straight tension tests and for torsion shear tests. In tension, such a joint is much stronger than a bar of solder of equal cross-section. The reason lies in the fact that the solder in the joint is restrained from contracting laterally while extending. In torsion shear tests of such joints the distribution of shear stress across the joint is not accurately known. Therefore, it seems inadvisable to state the results of such tests in terms of shear stress. The difficulty could be minimized by using tubes instead of rods.

FIG. 1.—Coppet Strips Joined by Solder G and Pulled Apart in the Tension Testing Machine.

The parts are shown replaced in the approximate position occupied at the moment of failure (× 2).

Chadwick (**5**) used a special type of butt joint consisting of a flattened loop of copper strip, soldered together with the free ends bent perpendicular to the flattened loop. The joint was tested by gripping the free ends and pulling at a constant speed. Failure occurred by a progressive tearing action at a load which remained steady after the failure had started. The results could not be expressed in terms of load per unit area. The tearing load was proportional to the width of the strip.

Simple lap joints are subject to bending in the strip near the joint when pulled in a tension testing machine. The eccentricity of loading and the bending may be expected to result in lower values of breaking load per unit lap area than if no bending or tension components were present. Gonser and Heath (**6**) and Dowdell, Fine, Elliot, and Mattson (**7**) reported results of tests on single lap joints in terms of shear stress.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 612.

In a single lap joint having a strength greater than the load required to produce considerable bending and yielding in the strip, failure starts by cracking at one or both ends followed by gradual tearing. Figure 1 is a photograph of a copper-to-copper soldered lap joint after the joint was broken and the parts fitted together in the approximate position occupied at the moment of failure. It seems advisable to state the strength of such a joint simply as the maximum load supported. All pertinent dimensions and testing conditions should be noted.

In spite of the disadvantages accruing to the use of the single lap joint, it was used for the most part in the present investigation for the following reasons: (1) the simple lap is representative of a large number of joints in use; (2) the sheet and strip materials were readily available, whereas materials for butt and sleeve joints were not; and (3) the labor and shop facilities at hand made the use of simple lap joints relatively convenient.

In any type of soldered joint, yielding of the material adjacent to the joint may result in a lowered strength per unit area of joint. For any given size of material and type of joint, a higher load per unit lap area would be supported by a short joint than by a long joint where appreciable yielding occurs.

Materials, and Conditions of Soldering and Testing:

A number of variables greatly influence the strength of soldered joints, and great care must be exercised in order to achieve reproducibility of test results. Discussions of the effect of the composition of the materials joined have been given in the literature (1,2,5,6); however, there has apparently been no very complete study of how and why the strength depends on the different materials joined.

Other factors which must be considered in making and testing soldered joints are the effects attributable to the fillet, the soundness of the joint, the rate of loading; the temperature during soldering, aging, and testing; the film thickness, fluxing, the "workability" of the solder; and pretinning or any other pretreatment of the surfaces. If a standardization of test procedure were to be made, all of these factors in addition to the type of joint and materials joined would need to be specified.

A fillet of solder may be expected to contribute to the strength of the joint and its contribution should not be included if the result is to be expressed as maximum shear stress within the lap.

Large bubbles or flux inclusions make a joint weaker than it would otherwise be. The technique of soldering can usually be regulated to make their effect negligible.

The speed of pulling has a profound effect on the maximum shear stress obtained in a soft soldered joint. For example, Maupin and Swanger (2) reported a shear strength of 5500 psi. for a pulling speed of 0.06 in. per min. but only 235 psi., for a test lasting over 7000 hr. In that particular case the joints were copper-to-copper joined with 50–50 solder and tested at room temperature. It was their opinion that only from the results of long-time tests can the strength of soft-soldered joints be correctly evaluated.

Effects of soldering, aging, and testing temperatures on the strength of soldered joints have been discussed in the literature (1,2,3,5,8). As a rule, the higher the testing temperature, the weaker is the joint.

The choice of a proper flux depends on the materials joined, the service conditions to be encountered, and the required range of soldering temperatures. The action of fluxes has been discussed by Coffman and Parr (9), by Dean and Wilson (10), and by Seckerson (11).

It has been demonstrated (2,3) that the thickness of solder film providing maximum strength is in the range from 0.002 to 0.005 in.

The greatest disadvantage in using tin-lead solders containing 20 per cent or less tin lies in their relatively inferior "workability." Flow temperatures are so high that copper bits oxidize rapidly and the solders dross more readily than lower melting solders. These tendencies impose a rather severe upper limit on the allowable range of temperatures in which the solder can be used for bit and flame soldering. In dip soldering the restriction to a narrow range of working temperatures is not so inconvenient.

For tin-lead alloys containing less than eutectic proportions of tin but more than about 15 per cent tin, the lower the tin content the longer is the melting range. Below about 15 per cent tin, however, the melting range narrows with decreasing tin content. The liquidus temperature increases uniformly as the tin content is lowered below the eutectic amount (62 per cent). In running long seams by hand in sheet metal, a long pasty range is conducive to cracking while cooling. In spite of this, joints can be made with care and patience.

In some cases, pretinning the surfaces before final joining is known to produce stronger joints than if the solder is merely flowed into the joint (2,6); however, it is often not practical to pretin the surfaces in service applications.

Tinning Properties of Solder:

It would be very desirable to have a simple, reliable test for estimating the suitability of a solder for a specific job. A popular test presumably intended for this purpose is the simple spreading, or spot test. The interpretation of the results of such a test is not simple. In order to fill a joint properly a solder must (*a*) wet the surface and alloy readily with the material, (*b*) flow easily, and (*c*) have enough surface tension to pull itself quickly through small spaces, often against gravity. The area of spread on an open plate cannot be used to evaluate separately the degree to which any one of these required properties is possessed by the solder. Studies of surface tension, viscosity, and alloying and diffusion rates would give valuable information but would require much time and effort. Surface tension measurements for some low melting metals and some tin-lead alloys have been reported (9,12,13). Sufficient data on the viscosity of molten solders and on the alloying reaction rates and diffusion rates of tin in other metals are lacking. In the absence of such data, it seems that the next best thing that one can accomplish quickly is to study the effect of temperature on the spread of different solders on the various metals to be soldered.

MATERIALS, TESTS, AND TEST SPECIMENS

Solders:

Six solders were investigated. Of these, four corresponded in composition with solders B, D, E, and G included in Federal Specification for Solder; Tin-Lead, QQ-S-571a, dated February 11, 1942, and one corresponded in composition with solder M listed in Emergency Alternate Federal Specification for Solder; Tin-Lead, E-QQ-S-571a, dated September 29, 1942. In addition, a commercial 50–50 tin-lead solder was investigated. All of the solders, except flux-cored wire solder, of the two Federal specifications referred to are listed in Table II. The six solders investigated are listed separately in Table III. Solders F and H of Federal Specification QQ-S-571a were not investigated because they are not ordinarily classed as low-tin solders, and their use at present is very limited. Solder R of Emergency Alternate Federal Specification E-QQ-S-571a was not investigated because it was understood that the can-making industry has made an extensive investigation of this solder, and also because its use in general purpose handwork is hindered by the high liquidus temperature (579 F. (304 C.)). Maupin and Swanger (3) reported results of tests on solder R.

TABLE II.—COMPOSITIONS AND MELTING RANGES OF THE SOLDERS OF FEDERAL SPECIFICATIONS.

	Class	Composition by Weight, per cent											Approximate Melting Range,[c] deg. Fahr.	
		Tin plus lead, min.	Lead	Tin	Antimony, max. or range	Copper, max.	Iron, max.	Bismuth, max.	Zinc, max.	Aluminum, max.	Silver	Total Other Elements	Solidus	Liquidus
	A	99.6		49 to 51	0.12	0.08	0.01	0.10	0.005	0.005		0.08	360	420
	B	99.5		38 to 42	0.12	0.08	0.01	0.25	0.005	0.005		0.08	360	460
	D	97.6		34 to 36	0.75 to 1.50	0.08	0.02	0.025	0.005	0.005		0.50	360 to 365	490 to 500
a	E	98.3		28 to 32	0.75	0.15	0.02	0.25	0.005	0.005		0.50	360	500 to 510
	F	99.6		69 to 71	0.12	0.08	0.01	0.10	0.005	0.005		0.08	360	378
	G	98.3		18 to 22	0.75	0.15	0.02	0.25	0.005	0.005		0.50	360	525 to 545
	H	99.6		59 to 61	0.12	0.08	0.01	0.10	0.005	0.005		0.08	360	372
b	M		remainder	19.5 to 20.5	0.50	0.08	0.02	0. to 0.75	0.005	0.005	1.15 to 1.35	0.08	355	518
	R		remainder	. . .	. . .	0.08	. . .	. . .	. . .	. . .	2.40 to 2.60	0.05	579	579

[a] Taken from "Federal Specification for Solder; Tin-Lead" QQ-S-571a Feb. 11, 1942.
[b] Taken from "Emergency Alternate Federal Specification for Solder; Tin Lead" E-QQ-S-571a, Sept. 29, 1942.
[c] For information purposes only.

TABLE III.—COMPOSITIONS AND MELTING RANGES OF THE SOLDERS INVESTIGATED.

	Solder	Composition by Weight, per cent[c]						Melting Range[d]						Density at 24 C., g. per cu. cm.[e]
								Solidus Temperature		Temperature of Maximum Heat Evolution		Liquidus Temperature		
		Tin	Antimony	Copper	Bismuth	Silver	Lead	deg. Cent.	deg. Fahr.	deg. Cent.	deg. Fahr.	deg. Cent.	deg. Fahr.	
	50–50 commercial	[f]	[f]	[f]	[f]	[f]		182[g]	360[g]			217[g]	420[g]	8.85
	B	40	0.30	0.06	0.20	0	Remainder	181	358	182	360	250	482	9.35
a	D	35	1.00	0.06	0.20	0	Remainder	181	358	184	362	256	493	9.50
	E	30	0.60	0.10	0.20	0	Remainder	181	358	183	361	261	502	9.64
	G	20	0.56	0.10	0.20	0	Remainder	181	358	183	361	282	540	10.16
b	M	20	0.40	0.06	0.56	1.25	Remainder	176	349	178	352	273	523	10.16

[a] Corresponding to Federal Specification QQ-S-571a, Feb. 11, 1942.
[b] Corresponding to Federal Specification E-QQ-S-571a, Sept. 29, 1942.
[c] Determined from the weights of the constituents before melting.
[d] Determined by A. I. Dahl, Associate Physicist, National Bureau of Standards.
[e] Determined by Miss M. G. Blair, Junior Physicist, National Bureau of Standards.
[f] Not determined, but presumably conforming in composition with Class A of QQ-S-571a.
[g] From reference (**2**).

The main reason for including a commercial 50–50 solder was to furnish a basis for comparison. Considerable data have been published on the strengths of joints made with 50–50 tin-lead solder (**2**,**3**). No analysis was made of the commercial 50–50 solder used in this investigation; however, it was supposed to correspond to class A solder of Federal Specification QQ-S-571a. Class A solder, before the tin shortage, was widely used in bit soldering and for sweated joints in plain, tinned, and galvanized steel stock and for copper and copper alloys. Class B solder was used for the same purpose as A where speed and "workability" were not so important. It was also used for dip soldering. Class D solder has been the customary plumber's wiping solder. It has recently been demonstrated (**14**) that perfect wiped joints with large savings in tin can be effected by a change from the old technique of wiping. Class E solder is intended mainly for filling dents as on automobile bodies. Class G solder is used for automobile work and for general purposes where a low tin content may be used. Class M, like G, contains 20 per cent tin and is intended for general soldering purposes.

Materials Joined:

The following sheet and strip materials were used:

1. Tin-plated mild steel, total thickness, 0.032 in.
2. Galvanized, sheet iron, total thickness, 0.065 in.
3. Black iron, thickness, 0.061 in.
4. Black iron, thickness, 0.070 in.
5. Yellow brass, thickness, 0.0625 in.
6. Yellow brass, thickness, 0.125 in.
7. Copper, hard rolled, thickness, 0.094 in.

Soldering Procedure:

In making the lap joints, the two strips to be soldered were laid in a horizontal position on a simple jig. The ends of a chromel-alumel thermocouple were silver-soldered into a silver button located directly below the joint and in direct thermal contact with the lower piece. The button was protected from the flux and solder by a sheet of mica from 0.001 to 0.003 in. thick. The surfaces to be soldered were cleaned by abrasion, and a saturated aqueous solution of a eutectic mixture of zinc and ammonium chlorides was applied to both surfaces. The strips were placed in position on the jig, so that the only force holding the specimens together was that of gravity. A short length of the solder wire was laid close to the entrance of the joint and a gas-air flame was applied to the top surface of the overlying strip. As soon as the desired temperature (usually 50 C. above the liquidus temperature) was reached, the joint was cooled by gently applying a wet cloth. The joints were washed clean and dried immediately after removal from the jig. No difficulty was encountered in getting any of the solders to flow and completely fill the joint. Almost all joints contained trapped gas bubbles. All six solders were about the same in this respect. A rough estimate of the area covered by bubbles was made for every joint after testing. It was found that as long as the bubbles were 1 mm. or less in diameter and occupied in aggregate less than 10 per cent of the area of the lap, there was no discernible weakening of the joint on this account. Enough joints were made practically 100 per cent free from bubbles to draw this conclusion. A small amount of manipulation of the parts of the joint was helpful in eliminating large bubbles, particularly from laps having areas of 1 sq. in. or more. It was also helpful in this respect to raise the temperature of the joint to 50 C. above the liquidus point rather than soldering at or near the liquidus point. The reason for this was partly due to the extra time during which bubbles escaped and partly due to the greater volume of the bubbles at the higher temperature.

Tension Testing Procedure:

Measurements were made to determine the solder film thickness and the area of lap. Areas occupied by fillets were not included. The specimens were pulled in a 10,000-lb. tension testing machine, on which the load was measured by a system of levers and counterpoises. The speed of the crosshead was 0.06 in. per min. for all the tests. Wedge grips were used.

The Spot-Testing Procedure:

Pieces of sheet iron and copper 3 to 4 in. square were cleaned with HCl. Equal volumes (0.05 cu. cm.) of different solders were placed at the center. Ten drops of saturated solution of zinc and ammonium chlorides in eutectic proportions were placed on and around the solder. The specimens were then placed in a preheated oven and held for different times and at different temperatures. On removal, the specimens were cooled without disturbing the solder. The specimens were then washed and the areas measured by means of a planimeter.

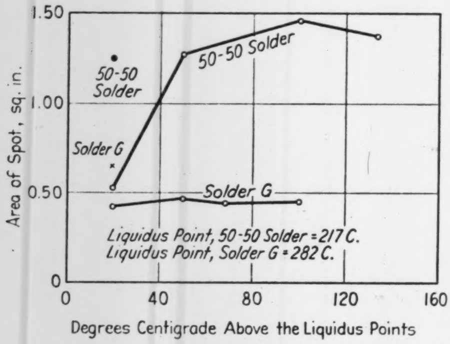

FIG. 2.—Spot or Spreading Tests Made on Copper Sheet Cleaned with HCl.

The two points not on the curves are results obtained on sheets roughened by scratching with a coarse file. Flux: eutectic $ZnCl_2 + NH_4Cl$. Each point plotted is the average of four determinations.

RESULTS

Compositions, Melting Ranges, and Densities of Solders:

The ranges of composition and the approximate melting ranges of the various solders listed in Federal Specifications QQ-S-571a and E-QQ-S-571a are given in Table II. The melting range for the 50–50 tin-lead solder is taken from a recent publication (2). The compositions, the melting ranges, and the densities of the actual solders used are given in Table III. The values of density were used in measuring out equal volumes of solders for the spot tests. Because of the difference in densities, the mass of solder G or M required to fill a given space or joint is approximately 15 per cent greater than that of 50–50 solder.

Spot Tests:

Effects of Temperature, Time, and Sheet Material.—The results of the spot tests on copper with solder G and 50–50 solder are given in Fig. 2. The area of the spots is plotted as a function of the number of degrees Centigrade above the liquidus temperature. The specimens were kept in the oven for 10 min. after the solder had completely melted. The temperatures measured were those of the oven. One twentieth of a cubic centimeter of solder was used for each spot. Only data for solders G and 50–50 are shown since they illustrate the type of result obtained. The curve for solder M, if plotted, would lie slightly above that for G. The curves for the other solders would be above that for solder M, but below the curve for 50–50. The tin content seemed to be a

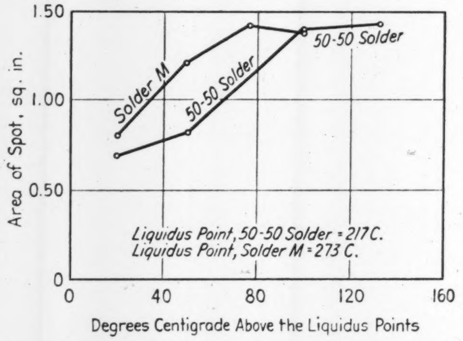

FIG. 3.—Spot or Spreading Tests Made on Black Iron Cleaned with HCl.

Flux: eutectic $ZnCl_2 + NH_4Cl$. Each point plotted is the average of four determinations.

fair qualitative index of the spreading of the solder on copper. On black iron, however, this was not the case.

A comparison of the results of the spot tests on black iron with solders M and 50–50 is given in Fig. 3. It was found that the solder having the lower tin content spread farther on black iron than the one with higher tin content. This is contrary to the results obtained on copper. The curve for solder G would be very much the same as that for M. The curves, if plotted for the rest of the six solders, would lie between the two curves shown. The area of the spots obtained by holding the sample of black iron at a given temperature for 1 hr. was not appreciably greater than that obtained at the same temperature for 10 min. On copper, however, there was generally a greater spread after 1 hr. than after 10 min.; the higher tin solders showed the greater increase in this respect. The spot tests in which the solder was held above the liquidus temperature for 1 hr. do not approach the condition of any actual soldering operation; they were made merely to show the effect of time upon the spreading of the solder. The results represented by each point in Figs. 2 and 3 are the average of four observations. The usual ratio of the maximum to minimum value for any given set of conditions was about 3 to 2.

TABLE IV.—AVERAGE STRENGTH OF LAP JOINTS 1 IN. WIDE WITH FILLETS.

Sheet Metal	Lap Length, in.	Breaking Load, lb.					
		Solder G	Solder M	Solder D	Solder E	Solder B	50-50 Solder
SINGLE LAPS—AVERAGE OF FOUR SPECIMENS							
0.070 in. black iron	0.56	2720	. . .	. . .	. . .	. . .	. . .
0.062-in. black iron		2120	2150	2120	2100	2100	2100
0.070-in. black iron	0.28	1530	1850	1790	1700	1900	1550
0.062-in. black iron		1320	1400	1500	1460	1580	1400
⅛-in. brass	0.56	2600	2560	2400	2310	2360	2700
¹⁄₁₆-in. brass		2690	2610	2590	2650	2510	2570
⅛-in. brass	0.28	1520	1360	1390	1290	1460	1410
¹⁄₁₆-in. brass		1450	1370	1320	1410	1270	1450
0.065-in. galvanized iron	0.56	1750	1780	1980	2070	1800	2020
0.065-in. galvanized iron	0.28	1240	1260	1390	1450	1530	1370
0.094-in. copper	0.56	1700	1630	1690	1710	1770	1750
0.125-in. copper	0.28	. . .	. . .	. . .	. . .	. . .	1640
0.094-in. copper		1190	1240	1380	1310	1350	1310
0.032-in. tin plate	0.28	1010	960	1010	1000	970	1070
DOUBLE LAPS—AVERAGE OF TWO SPECIMENS							
⅛-in. brass	0.56	4320	4570	4960	5020	5360	5490
¹⁄₁₆-in. brass		3110	3230	3310	3360	3150	3300
⅛-in. brass	0.28	2650	2770	2950	2610	2760	2970
¹⁄₁₆-in. brass		2580	2590	2720	2590	2600	2680

Surface Roughness of Sheet Metal, and Flux.—In order to obtain maximum reproducibility of spot test results, it is essential to have the metal surfaces sufficiently plane and level to prevent spreading by running down hill to one side of the sheet. Furthermore, the surface finish should be standardized, at least on a given material, since surface roughnesses, particularly scratches, promote spreading (see the points off the curves, Fig. 2). The spot tests were purposely made in air to simulate actual soldering conditions. Rhines and Anderson (**4**) used a shorter spreading time and obtained less coverage in proportion to the volume of solder. For the purpose of comparing the effectiveness of different fluxes to promote spreading, Coffman and Parr (**9**) and Dean and Wilson (**10**) used closed systems containing only the flux or gas under investigation.

Strength of Joints:

Solder Composition and Metals Joined.—In the short-time strength tests under consideration (2 to 9 min.) there was, for most materials, little difference between the results for single lap joints made with the six solders on any one material. The results of the observations for single and double lap joints are given in Table

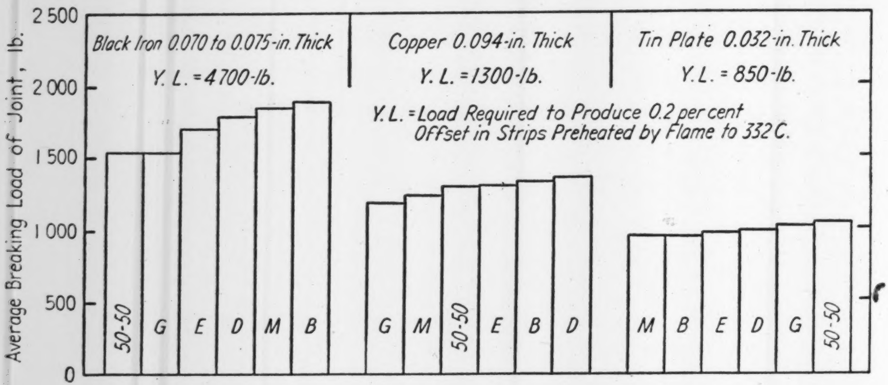

FIG. 4.—Short-Time Tests of Single Lap Joints Made with the Six Solders on Different Materials.

The lap length was 0.28 in. and small fillets were left on the work. Each value is the average of four determinations. Speed of moving crosshead = 0.06 in. per min. Room temperature.

IV. Because of the reasons previously discussed, it was considered inadvisable to express the strengths in terms of shear stress, consequently only the values for maximum load during test are given. Results of tests of single lap joints of black iron, copper, and tin plate made with each of the six solders are shown graphically in Fig. 4. It is evident that there is little, if any, correlation between the tin content of the solders and the strength of the joints represented in Fig. 4. A similar situation was found in the case of joints between brass strips $\frac{1}{8}$ in. thick, brass strips $\frac{1}{16}$ in. thick, and black iron strips 0.061 in. thick. The results obtained with galvanized iron were distinctive in that joints made with both solders G and M were noticeably weaker than joints made with any of the other solders. This difference ranged from 250 to 390 lb. (or about 20 to 30 per cent) for a 0.28-in. lap in strips 1 in. wide. In Fig. 4 it is evident that the breaking loads of the joints ranked in the same order as the loads necessary to produce 0.2 per cent offset in the strips at points well removed from the joint.

The joints in black iron strips 0.070 in. thick were stronger than similar ones in copper strips 0.094 in. thick, and these in turn were stronger than those in tin plate 0.032 in. thick as may be seen in Fig. 4. The lap length was the same for all tests, the results of which are given in the figure. How much of these differences was due to the bonding strengths and how much was due to the yielding of the strips during test was not determined. It is apparent from the graph that yielding in the tin and in the copper was probably of greater consequence in limiting the strength of the joint than was the case for black iron. The yield loads were determined on strips not soldered, but flame heated to 332 C. An example of how much the joint strength can depend on the yield strength of a material is pro-

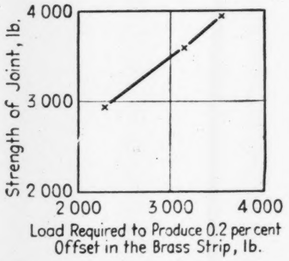

FIG. 5.—The Relation Between the Strength of Soldered Lap Joints in Yellow Brass Strips and the Load Required to Produce 0.2 per cent Offset in the Strips.

Dimensions of the strips: ⅛ by 1 by 6 in. Length of lap = 1.18 in. Solder G was used and a fillet was left on the work. Speed of moving crosshead = 0.06 in. per min. Room temperature.

vided in Fig. 5. The results represented in Fig. 5 were all for joints between strips of yellow brass 1 by 6 by $\frac{1}{16}$ in. Commercial yellow brass from different stocks having different tensile properties was used. The lap length was 1.18 in. and the solder used was solder G. The joint was long enough so that its strength exceeded the load required to produce 0.02 per cent offset in the materials soldered.

Size of Lap and Effect of Fillets.—In highly idealized experiments in which the forces can be maintained in pure shear and the fillets eliminated, the results are relatively easy to interpret in so far as comparisons of materials are concerned. In this study, however, it was desired to show what kind of results might be obtained under probable shop or field conditions. In making lap joints, the length of the lap is likely to vary, depending on the job, also the plumber or tinsmith doing hand soldering often cannot avoid leaving a fillet of solder on the work. An example of how much these variables can affect the strength of the joint is shown in Fig. 6 for joints made with solder G on $\frac{1}{8}$-in. thick brass. It is evident from the figure that an attempt to state the strength in terms of maximum shear stress is meaningless for short laps with solder fillets left on the work. Similar experiments carried out on the other strip and sheet materials included in this investigation showed considerable differences in the contribution by the fillet to the strength of a joint of a given lap length. In the softer, weaker materials, the contribution by the fillet to the strength was in general less than for the stronger materials. For example, the contribution by the fillet to the strength of joints in copper strips 0.094 in. thick was negligible for laps exceeding 0.5 in. in length. This was in contrast to the results on $\frac{1}{8}$-in. brass represented in Fig. 6. It is clear that yielding of the copper destroyed the holding strength of the fil-

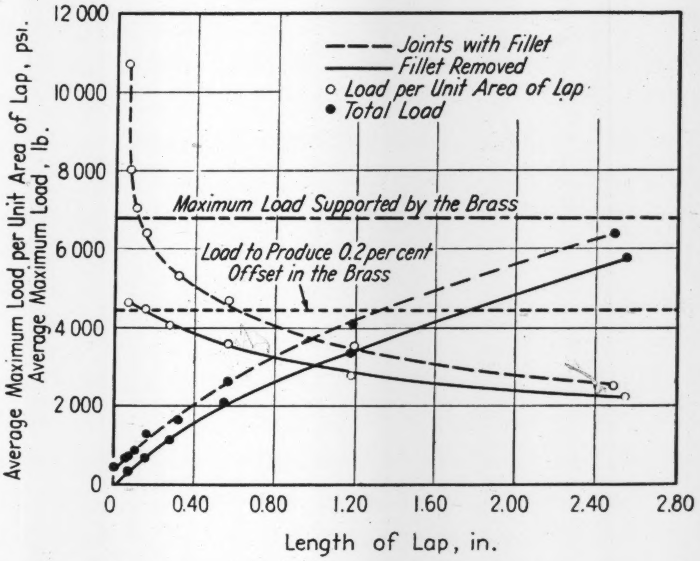

FIG. 6.—The Strengths of Single Lap Joints Between Brass Strips ⅛ by 1 in. in Section Jointed with Solder G.

Speed of moving head = 0.06 in. per min. Room temperature. Each point is the average of 4 specimens.

let before the maximum load was reached. In very long laps where failure starts by cracking at the ends and gradual peeling away ensues, it is meaningless to speak of the strength of the joint in terms of shear stress.

In all the experiments it was found that the solder left in fillets was not used to the best advantage. In the joints made with solder G on brass strips $\frac{1}{8}$ in. thick, the total strength of a 0.6-in. lap was about 2700 lb. on the average; of this only 500 lb. was contributed by the fillets, although there was, on the average, about twice as much solder in the fillets as in the joint. The fillets were approximately triangular in section with $\frac{1}{16}$-in. base and altitude. The solder film was from 0.002 to 0.004-in. thick.

Film Thickness.—The variation in the strength of lap joints made with solder G on brass strips $\frac{1}{8}$ in. thick is shown as a function of film thickness in Fig. 7. The optimum film thickness is of particular interest in the case of low-tin solders because of the common impression that more solder is required to make joints with a 20 per cent tin solder than with 50–50 solder.

It may be noted that the maximum strength of the lap joints compared favorably, on an equal area basis, with that reported by Maupin and Swanger (2) for copper sleeves joined with brass plugs by 50–50 solder. Nightingale and Hudson (1) found that the optimum film thickness depended on the soldering temperature. They attributed a large part of this dependence to the variation in fluidity and activity of the flux at various temperatures; however, their results and those of Maupin and Swanger (2) indicate that the optimum film thickness is in the range of 0.002 to 0.005 in. for solders containing approximately 50 per cent tin. Figure 7 shows that the optimum thickness of solder G on brass is from 0.002 to 0.004 in. The reason, if any exists, for using more of solder G than of 50–50 for a given joint consists of handling difficulties rather than the requirements of the joint.

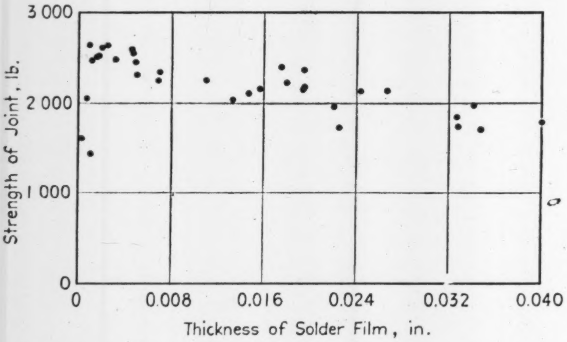

FIG. 7.—The Influence of Solder Film Thickness on the Strength of Single Lap Joints Between Brass Strips ⅛ by 1 in. in Section Jointed by Solder G.

The length of lap was 0.5 in. Speed of moving crosshead = 0.06 in. per min. Room temperature. Each point represents one specimen.

Other Factors Affecting the Strength of the Joint.—In the present investigation it was found that there was no significant difference between the sound joints made at the liquidus temperature and those made at 50 C. above the liquidus temperature. This was true for all six solders and all five sheet or strip materials joined. The soldering temperatures ranged from 217 to 332 C., depending on the solder. A saturated aqueous solution of a eutectic mixture of zinc and ammonium chlorides was used as flux and there was no difficulty in obtaining sufficient fluidity and activity of the flux at any of the temperatures used. At the higher temperatures it was necessary to exercise more care to avoid excessive drossing by oxidation of the liquid solder. High soldering temperatures may be expected to produce appreciable softening of some materials. For example, it was found that the ultimate tensile load that could be supported by the copper strips used in these experiments was lowered from an initial value of 5000 lb. for the 1 in. wide strip to 3200 lb. by heating with a flame to 50 C. above the liquidus of solder G. The lowering of yield load for 0.2 per cent offset was greater, being changed from 4500 lb. to only 1300 lb.

In the present experiments the temperature of the room ranged from extremes of 65 F. to 90 F. (18 C. to 32 C.) at different times during the tension tests. Maupin and Swanger (2) found that this temperature difference caused a difference of 900 psi. in the shear strength of copper to copper sleeve joints made with 50–50 solder, when pulled at the rate of 0.06 in. per min. In their tests this amounted to about 16 per cent of the average shear strength at room temperature.

The speed of pulling in the present experiments was standardized at 0.06 in. per min. for the speed of the moving crosshead. This was not necessarily the same as the speed of extension of the soldered joint. The time under load to fracture varied from a minimum of 2 min. to a maximum of 9 min. A proper selection of testing speed must depend on the correlation expected between service conditions and the test. The procedure must also be governed by the apparatus available. At present there seems to be a lack of instruments capable of automatically controlling the rate of extension between gage marks during test.

SUMMARY

1. Five solders conforming in composition with Federal Specifications QQ-S-571a and E-QQ-S-571a and a commercial 50–50 solder were studied to determine some of the limitations on their usefulness. Emphasis was placed on solders G and M having 20 per cent tin content.

2. The melting ranges and densities of the six solders were determined. Almost 15 per cent more by weight of either solder G or M than of 50–50 solder is required to fill a given space. The high liquidus temperatures and wide melting ranges of the lower tin solders complicate the technique of soldering but do not preclude their use for most handwork where 50–50 solder was formerly used.

3. Spot or spreading tests of the six solders were made on sheet copper and black iron. The effects of spreading time, temperature, and surface roughness were investigated. The solders having relatively high tin content spread farther on copper than solders with low tin content, but on black iron the solders having high tin content possessed no advantage in this respect. The spreading of all six solders on black iron was about the same as the highest result obtained on copper (with 50–50 solder). The higher rate of alloying and tin depletion on copper is believed to be largely responsible for the differences obtained on copper and black iron.

4. Interpretation of the spreading test results must be made in the light of a number of controlling factors, and each sheet material should be considered separately. The spot test is unsatisfactory for the purpose of furnishing a single index of the general utility of the solder.

5. The average strengths of lap joints in sheet and strip specimens made with solders having only 20 per cent tin appeared quite satisfactory for practical purposes.

6. In short-time tests of single lap joints there was little difference in the strengths of joints in any one material made with the six different solders.

7. It was evident that shear strength is not always the proper term to use in evaluating the strength of a single lap joint as ordinarily made in the shop or field.

8. A number of factors influence the results obtained in attempting to measure the strength per unit area of soldered joints. Among these are the type of joint; the length of lap; the fillet; the composition and strength of the metal joined; the soundness of the joint; the thickness of the solder film; the speed of testing; the temperatures of soldering, aging, and testing; and pretinning of the surfaces to be soldered. It is apparent that if some standard testing procedure were established, comparisons of results would be greatly facilitated. The proper testing procedure would be, of course, influenced by the desired correlation with practical applications and the facilities at hand.

Acknowledgment:

The authors gratefully acknowledge their indebtedness to Mrs. S. K. Gutterman whose excellent work in conducting many of the tests involved in the work has expedited the investigation and added very materially to whatever merit it may possess.

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DISCUSSION

Mr. Myron Park Davis.[1]—I should appreciate it, and I am of the opinion that many others would also appreciate it, if the authors would give us some detailed information about the fluxes that are used with these new solders.

This, I believe, is quite an important part of the soldering operation.

Mr. C. P. Xenis.[2]—One field that has not been mentioned during this discussion or in discussion of the paper by Colwell and Lang[3] is the subject of the "wipe" of lead sleeves on leaded cables, such as is used in splicing underground power cables and telephone cables.

In an attempt to reduce the amount of tin that is used in this type of work, extensive investigations were made by my company, the Consolidated Edison Company of New York, Inc., and some rather interesting results came out from this investigation. Some of these results confirm conclusions mentioned in these papers, and I merely wish to emphasize them.

The composition of solder commonly used in the past was 40 per cent tin, balance lead, and in some cases a higher tin content (50 per cent) was used in making these solder wipes on leaded cables. It was found that a satisfactory substitute composed of 30 per cent tin, 2 per cent antimony, and 68 per cent lead could be used on most applications.

The presence of antimony in the amounts indicated improves the physical properties of the solder, and after a certain amount of practice good results are obtained by the splicers. This solder must be applied very much faster than the higher tin content solders formerly used because of its limited plastic range.

Mr. J. R. Townsend.[4]—As the subject of wiped joint cables has been brought up, I should like to say I agree with the previous speaker that a good joint can be made with an alloy of $32\frac{1}{2}$ per cent tin with some antimony. A porous joint will result if less tin is used. However, it is possible to save a lot of tin in wiped joints by the use of a new joint that has been developed for telephone cable. The old joint was spliced by using a convex wipe. As a result of careful laboratory tests we can now make a joint in which the sleeve is beaten down and a concave wipe instead of a convex wipe can be put on, and the cable joint is just as strong for every purpose as it was before, and it results in a saving up to three fourths of wiped alloy formerly used. We call this the "Victory Joint."

Mr. J. A. Kies[5] (*author's closure, by letter*).—We do not have sufficient information upon which to base a choice of the best fluxes to use with the low-tin solders. The eutectic mixture of zinc and ammonium chlorides was found to work satisfactorily with all of the solders tested on the various sheet or strip materials included in this investigation. Chlorides are not permissible fluxes in certain applications where corrosion must be minimized. Rosin or rosin in alcohol is still the usual choice for such applications. Two recent papers of interest are "Non-Corrosive Soldering Fluxes" by H. Silman and W. Stein, *The Metal Industry* (London) Vol. 62, pp. 306–308, 325–327 (1943), and "Researches for Improved Solder Fluxes" by H. Peters and B. Gonser, *The Welding Journal*, Vol. 8, August, 1943, pp. 373S–374S. Several commercial fluxes on the market are advertised as being noncorrosive. There is need for quantitative test data on the relative corrosiveness of the various fluxes which are available. Unfortunately many of the fluxes proposed as noncorrosive are not very active as fluxes; others are satisfactory enough for 50-50 solder but give off objectionable vapors when heated to the high soldering temperatures of the low-tin solders. We hope to have more information on fluxes at a later date.

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[2] Electrical Engineering Dept., Consolidated Edison Co. of New York, Inc., New York, N. Y.

[3] D. L. Colwell and W. C. Lang, "Conservation of Tin in Soft Solders," ASTM Bulletin, No. 123, August, 1943, p. 37.

[4] Materials Standards Engineer, Bell Telephone Laboratories, Inc., New York, N. Y.

[5] Associate Metallurgist, National Bureau of Standards, Washington, D. C.

We have conducted no experiments on the making or testing of wiped joints. Those who have developed the design and technique for the new economical wiped joint are to be congratulated.

THE ATMOSPHERIC CORROSION OF COPPER

By A. W. Tracy,[1] D. H. Thompson,[1] and John R. Freeman, Jr.[1]

Synopsis

Data are given on an investigation of the corrosion of several brands of copper in the form of sheet and wire exposed to industrial, marine, and rural atmospheres over several periods up to 10 yr. Sheet metal specimens were also exposed to sea water at half tide for periods up to 2 yr.

The extent of corrosion was determined by changes in weight, tensile properties, and electrical resistance.

Differences in resistance to atmospheric corrosion of the several types of copper were found to be small and probably have no significance in relation to service life.

The results of many tests on the corrosion of copper by atmospheric exposure have been published in the past 30 yr. Vernon[2] concludes that the common impurities in copper are without measurable influence in the early stages of tarnish and patina formation, with the exception of arsenic which causes a more rapid darkening of the surface and a more rapid falling off of reflectivity.

Hudson[3] exposed wires 0.066 in. in diameter to atmospheric corrosion for 5 yr. at South Kensington, London, England, and found that both high-conductivity copper and arsenical copper were equally corroded, as indicated by electrical resistance measurements, but that arsenical copper was corroded more than high-conductivity copper when the rate of corrosion was determined by changes in breaking load.

The atmospheric tests made by the Society's Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys[4] include one type of tough-pitch copper and a phosphorus-deoxidized copper. After a 6-yr. exposure to industrial, marine, and rural atmospheres, changes in weight and in physical properties indicate no significant difference in the corrosion resistance of the two types of copper.

There has been no known study of the relative corrosion resistance of the several types of copper coming from different sources, although claims have been made of the superior corrosion resistance of some brands as compared to others. In fact, it has been stated that the corrosion resistance of the older copper roofs was due to impurities, and that the failure of some more recent copper roofs was due to the electrolytic copper being too pure.

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[1] Assistant Metallurgist, Research Assistant, and Technical Manager, respectively, The American Brass Co., Waterbury, Conn.

[2] W. H. J. Vernon, "First Experimental Report of the Atmospheric Corrosion Research Committee," British Non-Ferrous Metals Research Assn., *Transactions*, Faraday Soc., Vol. 19, pp. 839–900 (1923–1924).

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At the suggestion of the late W. H. Bassett, it was decided to make a fundamental study of the corrosion resistance of copper of various types. Several brands were selected from a group on which a study of physical properties had been made in 1914, and other brands were obtained in 1931. Some of the brands of copper tested are no longer commercially available, but it was thought worth while to include them in these tests. Tests were started first on wire specimens, and in the spring of 1932 similar tests were started on sheet metal specimens. Relative corrosion resistance was determined by changes in weight, tensile strength, and electrical resistivity after exposure to various atmospheres for periods up to 10 yr. to date.

The following brands of copper were available for the test:

Standard electrolytic, N.E.C.
Standard electrolytic, Chile.
Prime Lake, Tamarack.
Arsenical Lake, Tamarack.
Arsenical Lake, Superior.
Silver-bearing Lake, Franklin.
Phosphorus-deoxidized, electrolytic.
Fire-refined, Wallaroo.
Fire-refined, A.B.S.
Special silver-bearing, electrolytic.

Made by adding silver to pure electrolytic copper at the refinery.

Special arsenical, electrolytic.

Made by adding arsenic to pure electrolytic copper at the refinery.

The compositions for the above brands of copper are given in Table I.

TABLE I.—ANALYSES OF COPPERS.

	S = Sheet W = Wire	Copper, per cent	Silver, per cent	Arsenic, per cent	Nickel, per cent	Antimony, per cent	Iron, per cent	Lead, per cent	Selenium, per cent	Tellurium, per cent	Sulfur, per cent	Oxygen, per cent	Zinc, per cent
No. 1—Electrolytic, N.E.C.	S W	99.9735	0.0005	0.0000	0.0000	0.0007	0.0031	0.0014	0.0000	0.0000	0.0029	0.0175	Trace
No. 1A—Electrolytic, Chile	S	99.963	0.0000	0.0004	0.0002	0.00034	0.0011	0.0002	0.0004	0.0004	0.0024	0.027+	
No. 1B—Electrolytic, Origin unknown	W	99.931	0.0039	0.0010	0.0010	0.0012	0.0020	0.0003	0.0010	0.0009	0.0068	0.0453	
No. 2—Prime Lake, Tamarack	S W	99.9528	0.0242	0.0007	0.0000	0.0003	0.0025	0.0008	0.0004	0.0005	0.0011	0.0167[a]	Trace
No. 3—Arsenical Lake, Tamarack	S W	99.3555	0.0225	0.563	0.0014	0.0030	0.0008	0.0023	. . .	. . .	0.0014	0.0501[a]	
No. 4—Arsenical Lake, Superior	S W	99.5602	0.0948	0.299	0.0018	0.0005	0.0003	0.0011	. . .	. . .	0.0022	0.0401[a]	
No. 5—Silver-bearing Lake, Franklin	S W	99.8762	0.0868	0.0014	0.0007	0.0001	0.0020	0.0003	0.0003	0.0005	0.0020	0.0297[a]	Trace
No. 8—Phosphorus-deoxidized, electrolytic	S W	99.9498	0.0002	0.0012	0.0014	0.0005	0.0080	0.0000	0.0005	0.0005	0.0075	0.000	Phosphorus 0.020
No. 9—Fire-refined, Wallaroo	S	99.692	0.0016	0.0027	0.235	0.0003	0.0029	0.0018	0.0100	0.0053	0.0021	0.0526	
	W	99.672	0.0068	0.0032	0.240	0.0004	0.0014	0.0008	0.0096	0.0012	0.0043	0.0654	
No. 10—Fire-refined, A.B.S.	S	99.900	0.0074	0.0157	0.0302	0.0050	0.0016	0.0008	0.0120	0.0016	0.0041	0.0418	
	W	99.900	0.0083	0.0080	0.0360	0.0027	0.0012	0.0012	0.0203	0.0014	0.0054	0.0408	
No. 11—Special silver-bearing, electrolytic	S	99.9130	0.068	0.0010	0.0034	0.0003	0.0014	0.0006	0.0002	0.0006	0.0015	0.0274	
	W	99.8990	0.065	0.0007	0.0032	0.0003	0.0013	0.0008	0.0003	0.0004	0.0019	0.0244	
No. 12—Special arsenical, electrolytic	S W	99.6408	0.0016	0.344	0.0030	0.0004	0.0013	0.0004	. . .	. . .	. . .	0.0085[a]	

[a] By difference.

The copper wires were hard-drawn from 0.363 to 0.1285 in. in diameter and tested in that condition. Three wires, 15 in. long, of each type or brand of copper, were placed in metal frames as

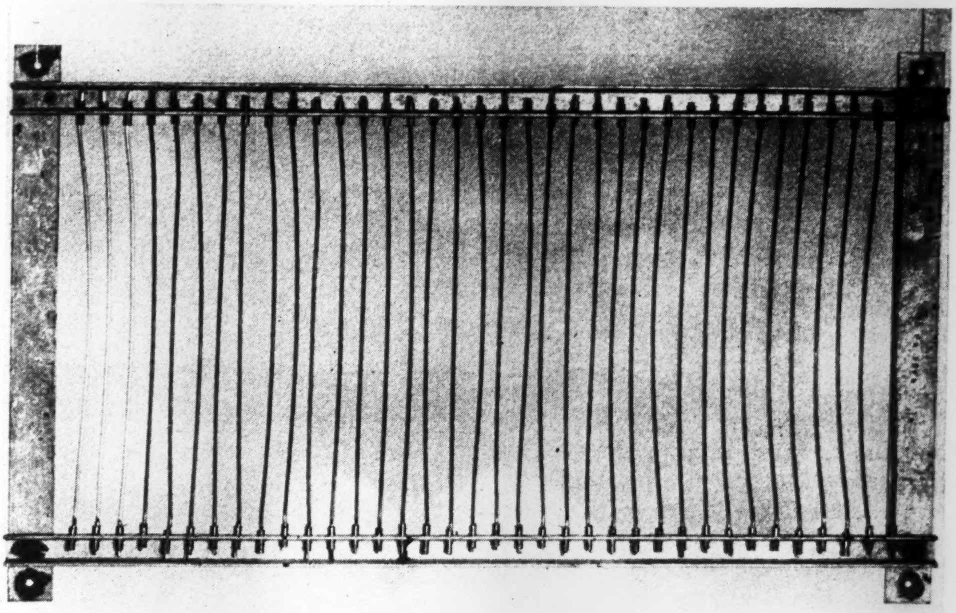


FIG. 1.—Wire Specimens in Exposure Frame.

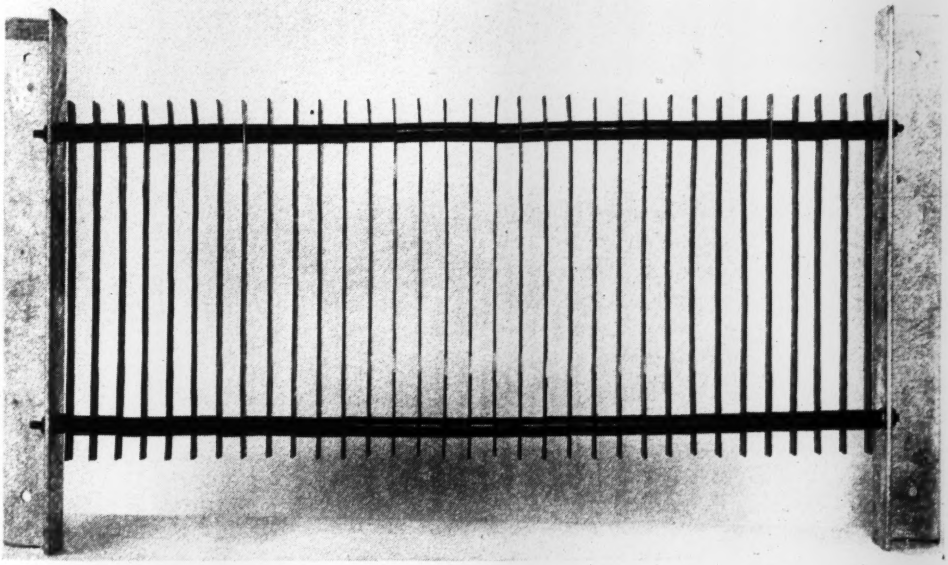


FIG. 2.—Sheet Metal Specimens in Exposure Frame.

shown in Fig. 1. The frames were mounted flat with all specimens inserted in them with the natural curvature of the coils downward, so that dew and rain water would collect at the mid-section of each wire to accelerate corrosive effects. They were insulated from the frames with phenolic-resin fiber tubes.

Ten racks were exposed at each test site to permit removing specimens at intervals over a period of years.

The sheet metal was tested in the form of strips 8 in. long by $\frac{5}{8}$ in. wide by 0.050 in. thick, machined to a tension test section $\frac{1}{2}$ by 0.050 in. with a 2-in. gage length. The specimens were cut from cold-rolled and annealed sheets. Three specimens of each lot of copper were placed in metal frames as shown in Fig. 2. The brass support rods were insulated by a coating of Bakelite varnish, and specimens were separated from each other by $\frac{1}{2}$-in. lengths of phenol-fiber tubes. The racks were exposed flat, except in sea water, where they were on edge so that the length of the specimen was vertical. Ten racks were provided for each exposure, except the rural and sea water exposures where only five were used.

Exposure Sites:

For normal industrial weathering, the racks were placed on the roof of a 4-story factory building in Waterbury, Conn., adjacent to a steam railroad. For accelerated industrial weathering, the racks were placed on the same roof but were sprayed with city water for 2-min. periods four times daily on an average of 5 days a week. Figure 3 shows the test sites. The marine exposure site was on a small island, in Long Island Sound near Guilford, Conn., about 4 miles from the mainland. The racks were at an elevation of 40 ft. and within 200 ft. of the water in three directions. Racks for the sea water test were fastened at half tide level to a dock on the island, so that the specimens were immersed alternately in clean sea water. The rural test station was in Winchester, Conn., several miles from the nearest industrial town and at about 1300 ft. above sea level.

The specimens were started on test and removed at the following intervals:

SHEET METAL SPECIMENS.

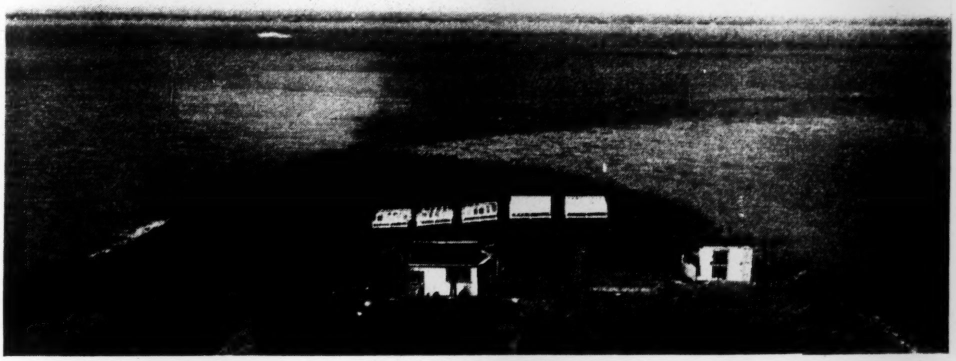
Nominal Exposure, yr.	Date Removed	Actual Exposure, yr.
NORMAL INDUSTRIAL WEATHERING, EXPOSED JUNE 2, 1932		
1	June 2, 1933	1.00
2	June 4, 1934	2.00
3	June 3, 1935	3.00
5	June 2, 1935	5.00
8	June 18, 1940	8.04
10	June 2, 1942	10.00
ACCELERATED INDUSTRIAL WEATHERING, EXPOSED JUNE 3, 1932		
1	June 2, 1933	1.00
2	June 4, 1934	2.00
3	June 3, 1935	3.00
5	June 2, 1937	5.00
8	June 18, 1940	8.04
10	June 3, 1942	10.00
MARINE ATMOSPHERE, EXPOSED MAY 25, 1932		
1	May 31, 1933	1.02
2	May 8, 1934	1.95
3	June 3, 1935	3.02
5	May 28, 1937	5.00
8	June 14, 1940	8.05
10	July 10, 1942	10.15
RURAL ATMOSPHERE, EXPOSED APRIL 28, 1932		
1	May 2, 1933	1.01
2	April 30, 1934	2.00
5	April 30, 1937	5.00
10	May 26, 1942	10.09
SEA WATER TESTS, EXPOSED MAY 25, 1932		
½	Dec. 5, 1932	0.53
1	May 31, 1933	1.02
2	May 8, 1934	1.95

NOTE.—The remaining two racks were lost in the great hurricane of September, 1938.

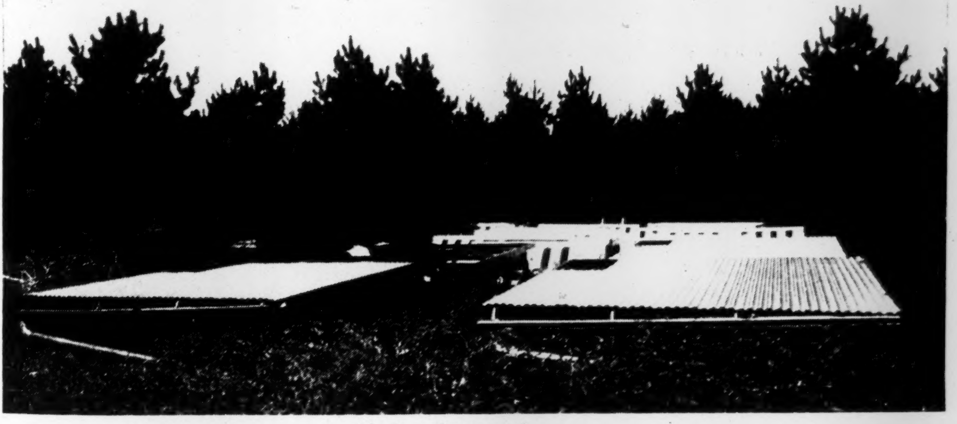
WIRE SPECIMENS.

Nominal Exposure, yr.	Date Removed	Actual Exposure, yr.
NORMAL AND ACCELERATED ATMOSPHERE, EXPOSED OCTOBER 7, 1931		
1	Oct. 7, 1932	1.00
2	Oct. 6, 1933	2.00
3	Oct. 8, 1934	3.00
5½	June 2, 1937	5.65
8½	June 18, 1940	8.70
10½	June 3, 1942	10.65
MARINE ATMOSPHERE, EXPOSED NOVEMBER 9, 1931		
1	Dec. 5, 1932	1.07
2	Nov. 20, 1933	2.03
3	Nov. 16, 1934	3.02
5½	May 28, 1937	5.55
8½	June 14, 1940	8.59
10½	July 10, 1942	10.68
RURAL ATMOSPHERE, EXPOSED NOVEMBER 4, 1931		
1	Dec. 22, 1932	1.13
2	Nov. 2, 1933	2.00
3	Oct. 19, 1934	2.96
5½	Apr. 30, 1937	5.48
8½	June 13, 1940	8.60
10½	May 26, 1942	10.56

(a) Industrial atmosphere.



(b) Marine atmosphere.

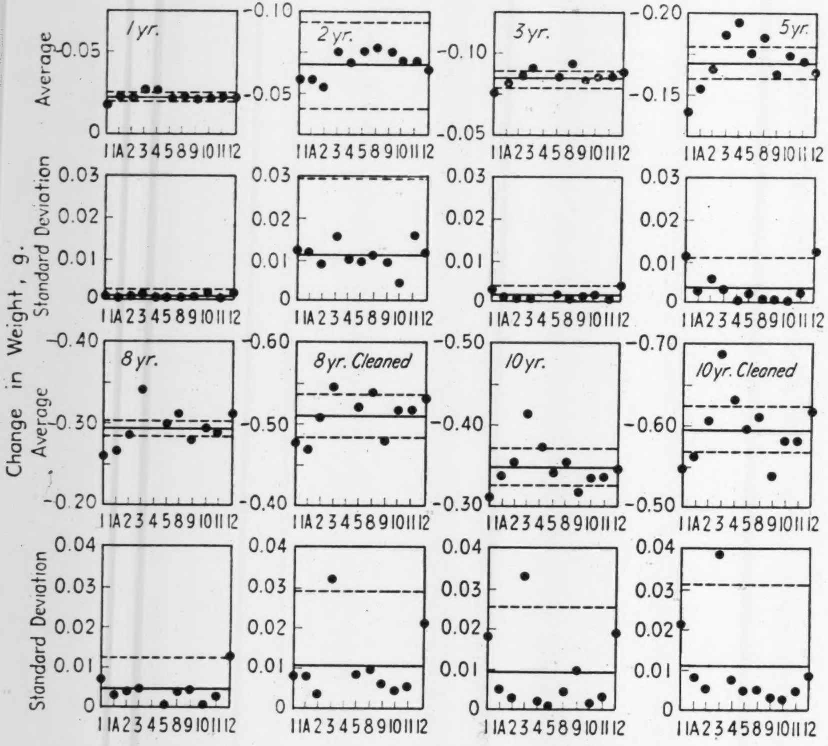


(c) Rural atmosphere.

FIG. 3.—Weather Exposure Sites.

SHEET METAL

The sheet metal tension specimens were weighed before and after exposure with a precision of at least 0.0005 g. The initial weights of the specimens were approximately 30 g. Specimens exposed to the several atmospheres were in all cases weighed without removing corrosion products. Specimens exposed 8 and 10 yr. were also weighed after removing corrosion products by a 2-min. dip in a cold 10 per cent sulfuric acid solution, scrubbing with a bristle brush under water, and drying. Specimens exposed to sea water were weighed only after removing the corrosion products. Tension tests for the original and all test periods, except the 10-yr. period, were made on a lever-balance tension machine having an accuracy of 1 per cent; the 10-yr. test specimens were pulled on a hydraulically-balanced machine with an accuracy of $\frac{1}{2}$ per cent. Elongation measurements had an accuracy of $\frac{1}{2}$ per cent.

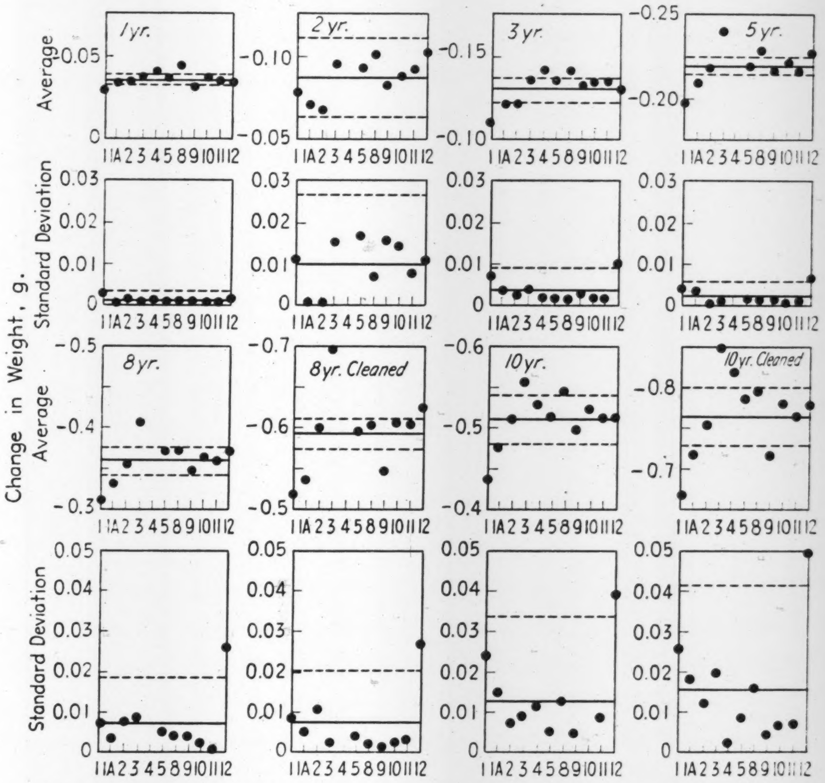


No. 1 Electrolytic, N.E.C.
No. 1A Electrolytic, Chile.
No. 2 Prime Lake, Tamarack.
No. 3 Arsenical Lake, Tamarack.
No. 4 Arsenical Lake, Superior.
No. 5 Silver-bearing Lake, Franklin.
No. 8 Phosphorus-deoxidized, electrolytic.
No. 9 Fire-refined, Wallaroo.
No. 10 Fire-refined, A.B.S.
No. 11 Special silver-bearing, electrolytic.
No. 12 Special arsenical, electrolytic.

FIG. 4.—Control Charts for Total Weight Changes, Sheet Metal in Normal Industrial Weathering.

Control charts based on average weight changes and standard deviations are shown in Figs. 4 to 8. It is appreciated that subgroups of three specimens are small for a formal statistical analysis, but it was considered that such a study would yield more information from the data than a general estimate as to the significance of the corrosion losses. The standard deviation,[5] σ, is the square root of the average of the squares of the deviations of the measurements from their average, $\bar{X}$. The control limits for averages were established from $\bar{\bar{X}} \pm 3 \frac{\bar{\sigma}}{C_2\sqrt{n}}$ where $\bar{\bar{X}}$ equals the grand average of all the weight changes at one test site for any period; $\bar{\sigma}$ equals a similar average of the standard deviations; C_2 equals 0.7236; and n equals 3, the number of specimens. Any averages outside these limits indicate differences in corrosion due to an assignable cause—in the present case, variation in composition.

The standard deviation charts have the average standard deviation of any group as the central line, and the limits are $\bar{\sigma} \pm 3 \frac{\bar{\sigma}}{C_2\sqrt{2n}}$. Any point falling



No. 1 Electrolytic, N.E.C.
No. 1A Electrolytic, Chile.
No. 2 Prime Lake, Tamarack.
No. 3 Arsenical Lake, Tamarack.
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No. 10 Fire-refined, A.B.S.
No. 11 Special silver-bearing, electrolytic.
No. 12 Special arsenical, electrolytic.

FIG. 5.—Control Charts for Total Weight Changes, Sheet Metal in Accelerated Industrial Weathering.

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[5] A.S.T.M. Manual on Presentation of Data, Am. Soc. Testing Mats., Philadelphia, Pa. (1940). (Issued as separate publication.)

outside the control limits for standard deviation indicates that the group of specimens represented by that point is not in statistical control.

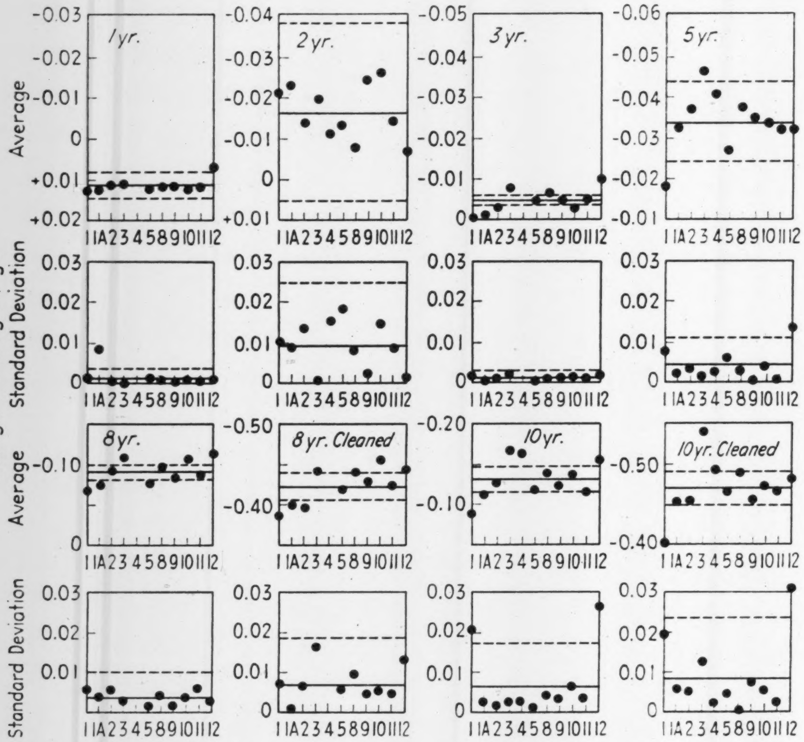
No method is known for determining similar control limits for data obtained from tension and elongation tests where measurements before and after exposure are made on different groups of specimens. Whether or not the tension test data showed significant corrosion losses with time was indicated if the tensile strength of either the original or corroded specimens fell outside the following limits:

$$\frac{\bar{X}_o + \bar{X}_c}{2} \pm \frac{\sigma_o + \sigma_c}{2} \cdot \frac{3}{C_2\sqrt{n}}$$

where:

$\bar{X}_o$ and σ_o = the average and standard deviation of uncorroded specimens, and

$\bar{X}_c$ and σ_c = the same statistics for the corroded specimens.



No. 1	Electrolytic, N.E.C.
No. 1A	Electrolytic, Chile.
No. 2	Prime Lake, Tamarack.
No. 3	Arsenical Lake, Tamarack.
No. 4	Arsenical Lake, Superior.
No. 5	Silver-bearing Lake, Franklin.
No. 8	Phosphorus-deoxidized, electrolytic.
No. 9	Fire-refined, Wallaroo.
No. 10	Fire-refined, A.B.S.
No. 11	Special silver-bearing, electrolytic.
No. 12	Special arsenical, electrolytic.

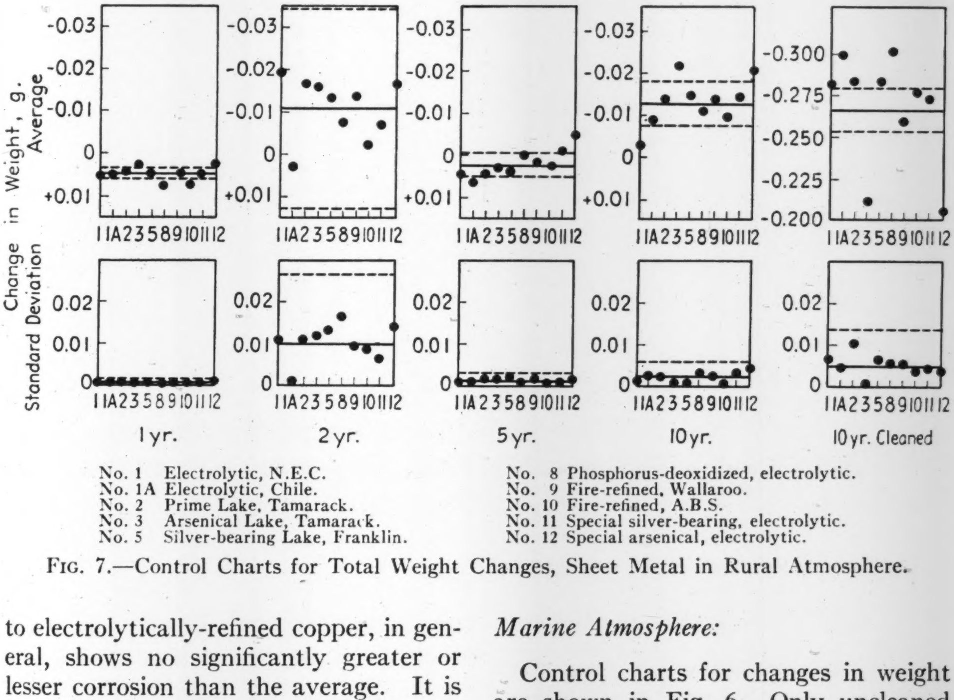
FIG. 6.—Control Charts for Total Weight Changes, Sheet Metal in Marine Atmosphere.

Normal Industrial Atmosphere:

It will be noted in Fig. 4, which shows the control charts for normal industrial weathering, that electrolytic copper, No. 1, and special arsenical electrolytic copper, No. 12, in general, show large standard deviations. These groups of specimens were at the ends of the test racks where one specimen of each group might have been partially sheltered by the end of the rack. The standard deviations of arsenical lake copper, No. 3, also tend to be high, but no explanation is evident. It is clear from an inspection of the diagram of averages that the two electrolytic coppers, Nos. 1 and 1*A*, and fire-refined Wallaroo copper, No. 9, were corroded significantly less than the average, and that the arsenical lake coppers, Nos. 3 and 4, were corroded significantly more than the average. Arsenical copper, made by adding arsenic to electrolytically-refined copper, in general, shows no significantly greater or lesser corrosion than the average. It is interesting to note that removing the corrosion products did not alter to any great extent the relative position of the different groups.

Accelerated Industrial Atmosphere:

It is interesting to compare the results of tests in normal industrial atmosphere with those in accelerated industrial atmosphere as shown in Fig. 5. The corrosion losses are larger for the accelerated test and only the arsenical electrolytic copper specimens are out of control. The spread of the control lines from year to year is quite similar in the two sets of tests, indicating that whatever factor was influencing results was acting almost equally in both tests. As in the normal industrial atmospheric test, N.E.C. and Chile electrolytic coppers and fire-refined Wallaroo copper were corroded significantly less than the average, and arsenical lake copper was corroded more than the average.

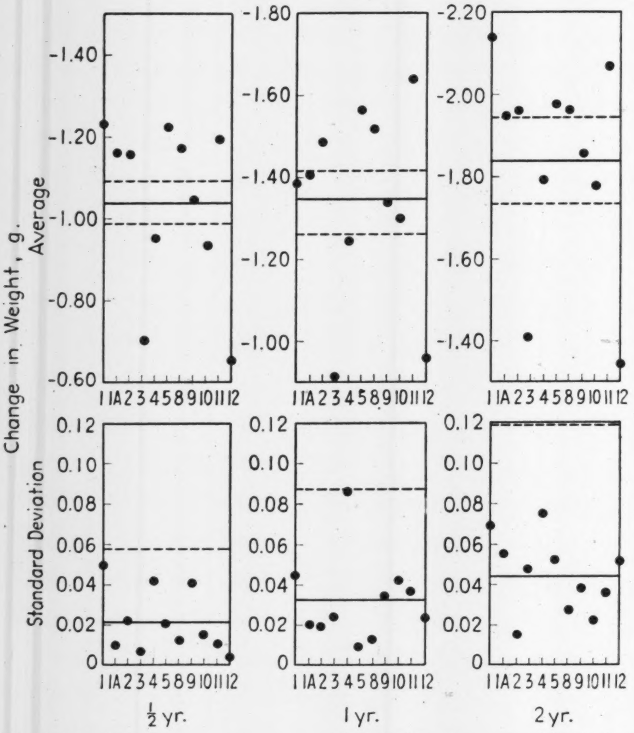


No. 1 Electrolytic, N.E.C.
No. 1A Electrolytic, Chile.
No. 2 Prime Lake, Tamarack.
No. 3 Arsenical Lake, Tamarack.
No. 5 Silver-bearing Lake, Franklin.
No. 8 Phosphorus-deoxidized, electrolytic.
No. 9 Fire-refined, Wallaroo.
No. 10 Fire-refined, A.B.S.
No. 11 Special silver-bearing, electrolytic.
No. 12 Special arsenical, electrolytic.

FIG. 7.—Control Charts for Total Weight Changes, Sheet Metal in Rural Atmosphere.

Marine Atmosphere:

Control charts for changes in weight are shown in Fig. 6. Only uncleaned specimens of electrolytic copper, Chile (No. 1*A*) in the 1-yr. test, electrolytic copper, N.E.C. (No. 1) in the 10-yr. test, and both cleaned and uncleaned specimens of arsenical electrolytic copper (No. 12) in the 5-yr. and 10-yr. test appear to be out of statistical control. During the first year, all types of copper gained weight, and only after 3 yr. were there significant differences in weight losses. There is some shifting in relative positions of the various types of copper in the several diagrams, possibly due to differences in the types of corrosion films formed. In general, it is evident that, as in the industrial atmospheres, pure electrolytic copper was corroded significantly less than the average, and arsenical copper significantly more than the average.



No. 1 Electrolytic, N.E.C.
No. 1A Electrolytic, Chile.
No. 2 Prime Lake, Tamarack.
No. 3 Arsenical Lake, Tamarack.
No. 4 Arsenical Lake, Superior.
No. 5 Silver-bearing Lake, Franklin.
No. 8 Phosphorus-deoxidized, electrolytic.
No. 9 Fire-refined, Wallaroo.
No. 10 Fire-refined, A.B.S.
No. 11 Special silver-bearing, electrolytic.
No. 12 Special arsenical, electrolytic.

FIG. 8.—Control Charts for Total Weight Changes, Sheet Metal in Sea Water, Half Tide Location.

Rural Atmosphere:

Figure 7 includes control charts for specimens corroded 1, 2, 5, and 10 yr. in a rural atmosphere. In these tests, all specimens were in statistical control, as indicated by the charts for standard deviation. Specimens exposed as long as 5 yr. to rural atmosphere and weighed without chemical cleaning, in most cases, still showed a gain in weight. The average weight loss of the uncleaned specimens at 10 yr. indicates that the several brands of copper show the same relative corrosion resistance among themselves as in the other atmospheres, although the weight losses are very low; that is, the electrolytic coppers were apparently corroded less than the average and the arsenical coppers were apparently corroded more than the average. When the corrosion products were removed chemically, the electrolytic, prime lake, silver-bearing lake, and deoxidized coppers had corroded significantly more than

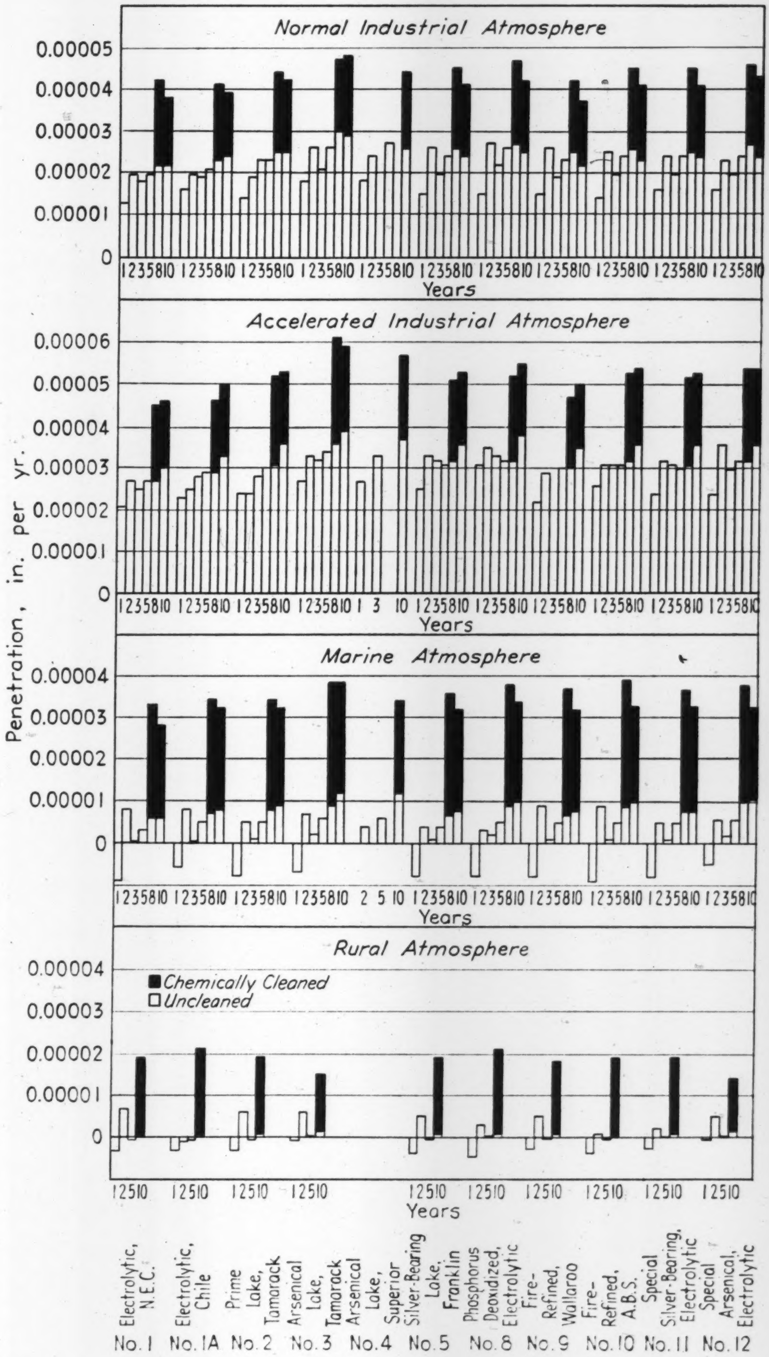


FIG. 9.—Penetration, Inches per year from Weight Losses.

the average, and the arsenical coppers had corroded significantly less than the average. The explanation for this anomaly is not apparent.

It has already been observed that the 2-yr. tests in all atmospheres showed wide spreads between the lines marking the control limits. A possible explanation is that the rates of film formation and film dissolving were not at equilibrium at that time. Opposed to this is the possibility that this point of non-equilibrium would not necessarily occur at 2 yr. at all the test locations.

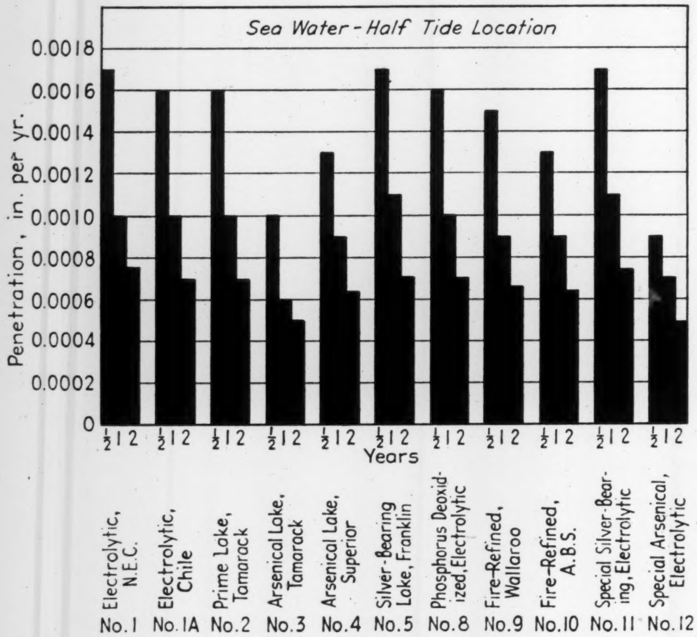


FIG. 10.—Penetration, Inches per year from Weight Losses, Sheet Metal in Sea Water, Half Tide Location.

Sea Water:

The tests in sea water, as previously mentioned, were made at half tide. Control charts prepared from the losses in weight are given in Fig. 8. None of the individual groups lies outside the control limit in the standard deviation charts. It will be noted that the high-arsenical lake copper (No. 3) and the arsenical electrolytic copper (No. 12) are very definitely more corrosion resistant than the other coppers. The electrolytic, silver bearing, and deoxidized coppers all corroded significantly more than the average.

Corrosion Rates:

Figures 9 and 10 show the average penetration in inches per year based on weight changes for the various types of copper in the several atmospheres. In the marine and rural atmospheres, many of the specimens gained in weight, and the penetrations are hypothetical figures. The unshaded areas represent penetrations in inches per year based on the weight losses of specimens from which corrosion products had not been removed.

The shaded areas are based on weight losses of specimens from which corrosion products were removed in a 10 per cent solution of sulfuric acid at room temperature. The upper set of graphs in Fig. 9 indicates that corrosion rates in the normal industrial atmosphere reached their highest point at 8 yr., but the corrosion rates are still increasing at 10 yr. in the accelerated industrial atmosphere. A comparison of the graphs for normal industrial and accelerated industrial atmosphere in Fig. 9 shows good agreement among the relative corrosion resistances of the various types of copper.

The penetration diagrams for the tests at the marine and the rural locations are also shown in Fig. 9. Uncleaned specimens had either very low corrosion rates or hypothetical gains due to the presence of corrosion products. The charts show quite definitely that corrosion rates should be determined from changes in weight of specimens from which corrosion products have been removed, rather than from specimens weighed without removing corrosion products.

The most interesting relationship in the diagrams of penetration at the half tide location, Fig. 10, is the large decrease in corrosion rate at the 1- and 2-yr. periods, indicating that protective films were forming.

Tension Tests on Sheet Metal:

Averages and standard deviations of tensile strength measurements in pounds per square inch of uncorroded specimens and those exposed to the several atmospheres and sea water are given in Table II. All tensile strength values are based on areas of cross-section measured before exposing specimens. The average per cent changes in tensile strength are shown in bar charts in Figs. 11 and 12. There is no consistent significance in the extent of corrosion revealed by tension tests on specimens exposed to the several atmospheres. There is no plausible reason why tensile strength should show losses in 1 yr. and gains in the next except for lack of control in the testing procedure. The graphs are interesting in that they repeat, to some extent, the trends in the penetration diagrams. The arsenical lake coppers tended to have greater losses in tensile strength than the other coppers. For some unexplained reason, the tensile strength losses were, in most cases, greater after the second year than the third year, which confirms the weight changes.

Percentage changes in tensile strength of specimens tested in sea water are shown in Fig. 12. In general, the losses in tensile strength, of each type of copper, confirm the weight losses. There is no apparent reason, however, why some of the losses in tensile strength should be greater after 6 months of exposure than after 1 yr.

Ductility of Sheet Metal:

The average elongations in 2 in. and their standard deviations are given in Table II. An examination of the data discloses that no significant losses in ductility occurred even in the 10-yr. test in normal industrial atmosphere and only arsenical electrolytic copper, No. 12, lost ductility during the 10 yr. in the accelerated industrial atmosphere. The tests at the marine and rural locations show insignificant losses in ductility. Even in the sea water test, where corrosion losses were greatest, only one type of copper, No. 1A (Chile), was significantly affected and then only at the longest test period, 2 yr.

Surface Condition of Specimens:

Normal Industrial Weathering.—After 1 yr., the arsenical coppers, Nos. 3, 4, and 12, were dark brown, almost black,

TABLE II.—SHEET METAL SPECIMENS.

	Average ($\bar{X}$) and Standard Deviation (σ)	Tensile Strength, psi.							Elongation in 2 in., per cent						
		Original	1 yr.	2 yr.	3 yr.	5 yr.	8 yr.	10 yr.	Original	1 yr.	2 yr.	3 yr.	5 yr.	8 yr.	10 yr.
NORMAL INDUSTRIAL ATMOSPHERE															
No. 1—Electrolytic, N.E.C.	$\bar{X}$	33 760	33 940	33 580	34 220	33 030	33 730	33 300	42.1	43.2	41.7	41.5	43.2	42.7	42.0
	σ	141	98.8	332	75.9	287	200	0	2.40	1.93	3.68	0.409	1.18	1.25	3.34
No. 1A—Electrolytic, Chile	$\bar{X}$	32 060	32 210	31 690	32 280	31 800	31 990	31 530	42.8	43.8	41.5	41.8	42.7	41.2	39.7
	σ	248	123	62.4	215	245	284	94.3	0.898	3.07	0.708	2.72	2.90	1.31	1.18
No. 2—Prime Lake, Tamarack	$\bar{X}$	33 290	33 680	33 050	33 550	32 450	32 930	32 770	42.4	42.7	42.0	40.3	45.3	40.5	42.7
	σ	224	119	188	190	40.8	115	144	1.40	2.72	2.95	0.944	1.25	1.08	1.31
No. 3—Arsenical Lake, Tamarack	$\bar{X}$	35 580	35 580	35 030	35 130	34 550	34 780	34 400	44.0	41.7	44.3	43.8	44.2	44.2	44.7
	σ	369	165	314	158	178	47.1	108	1.78	1.55	2.06	1.89	2.32	2.63	0.624
No. 4—Arsenical Lake, Superior	$\bar{X}$	35 160	34 670	34 460	...	34 400	...	33 800	42.0	43.3	43.2	...	41.5	...	43.7
	σ	201	243	305	...	245	...	141	1.89	1.65	3.12	...	2.12	...	2.78
No. 5—Silver-bearing Lake, Franklin	$\bar{X}$	33 440	33 890	33 320	33 660	32 920	33 290	32 920	44.6	43.3	46.8	43.5	44.3	44.7	44.5
	σ	152	162	25.0	14.1	184	154	193	2.32	1.03	0.750	1.64	0.85	1.65	1.38
No. 8—Phosphorus-deoxidized, electrolytic	$\bar{X}$	34 250	34 220	33 900	34 520	34 050	33 350	33 370	40.8	40.2	37.8	40.3	36.8	37.3	37.8
	σ	307	281	151	75.9	70.8	243	520	1.94	1.55	2.75	0.472	2.78	0.236	0.472
No. 9—Fire-refined, Wallaroo	$\bar{X}$	35 390	35 640	34 860	35 660	35 030	34 770	34 880	44.2	43.7	44.0	42.5	41.0	45.0	42.3
	σ	330	152	40.3	96.3	94.3	60.2	155	2.53	2.25	0.408	1.78	2.55	0.817	1.55
No. 10—Fire-refined, A.B.S.	$\bar{X}$	32 640	32 450	31 870	32 530	31 980	31 990	31 870	40.2	39.2	40.8	42.0	43.8	43.5	42.0
	σ	213	131	377	307	62.4	506	386	1.07	2.39	4.88	2.86	0.236	1.87	1.08
No. 11—Special silver-bearing electrolytic	$\bar{X}$	33 420	33 670	32 840	33 900	33 200	33 200	32 920	39.7	39.0	40.0	41.5	38.3	37.0	40.2
	σ	304	139	121	78.5	141	142	23.6	2.88	1.47	1.08	0.688	0.850	5.72	2.25
No. 12—Special arsenical, electrolytic	$\bar{X}$	33 910	34 890	33 480	33 790	33 780	33 030	33 730	42.7	38.7	39.7	34.8	37.8	37.5	37.3
	σ	143	171	611	444	209	1020	232	2.48	5.80	3.93	3.28	0.236	4.25	1.89

ACCELERATED INDUSTRIAL ATMOSPHERE

No. 1—Electrolytic, N.E.C.	$\bar{X}$	33 760	34 100	33 590	34 280	33 250	33 470	32 820	42.1	44.8	43.5	43.5	46.0	45.0	44.2
	σ	141	130	226	290	70.7	245	165	2.40	1.65	2.68	1.42	2.35	1.42	0.624
No. 1A—Electrolytic, Chile	$\bar{X}$	32 060	32 350	31 660	32 730	31 500	32 000	31 480	42.8	39.7	38.5	40.2	44.7	45.5	41.2
	σ	248	245	216	429	163	168	125	0.898	3.42	1.87	2.88	5.17	1.08	2.63
No. 2—Prime Lake, Tamarack	$\bar{X}$	33 290	33 280	32 610	33 600	32 800	32 490	32 330	42.4	42.8	41.5	40.5	40.5	46.5	42.0
	σ	224	177	186	42.3	147	41.9	103	1.40	0.624	0.408	1.08	1.78	1.47	1.08
No. 3—Arsenical Lake, Tamarack	$\bar{X}$	35 580	35 490	35 040	35 510	34 520	34 640	34 350	44.0	45.7	44.3	41.8	45.8	42.3	46.5
	σ	369	208	390	213	131	427	141	1.78	1.65	2.06	2.90	2.32	0.852	2.28
No. 4—Arsenical Lake, Superior	$\bar{X}$	35 160	35 070	...	34 880	...	...	33 820	42.0	44.2	...	41.7	...	...	41.7
	σ	201	236	...	77.2	...	...	275	1.89	3.48	...	2.02	...	...	2.62
No. 5—Silver-bearing Lake, Franklin	$\bar{X}$	33 440	33 260	33 080	33 400	32 770	32 910	32 530[a]	44.6	46.2	44.0	46.7	44.5	43.3	47.0
	σ	152	299	49.9	182	249	154	352	2.32	0.625	1.88	1.55	1.08	0.236	2.16
No. 8—Phosphorus-deoxidized, electrolytic	$\bar{X}$	34 250	34 590	33 510	34 160	33 730	33 720	33 950	40.8	38.2	40.0	38.3	37.8	39.0	40.5
	σ	307	489	321	363	419	416	426	1.94	1.31	2.04	0.625	1.55	1.63	1.08
No. 9—Fire-refined, Wallaroo	$\bar{X}$	35 390	35 500	35 130	35 690	34 820	35 090	34 820	44.2	44.7	45.2	42.5	44.8	44.0	43.3
	σ	330	144	106	53.5	225	235	125	2.53	1.55	2.02	1.87	1.88	2.16	0.944
No. 10—Fire-refined, A.B.S.	$\bar{X}$	32 640	32 610	32 050	32 150	31 820	31 630	31 600	40.2	43.0	41.0	39.7	40.3	41.8	40.3
	σ	213	439	569	1140	272	90.0	286	1.07	2.55	0	0.474	0.236	0.236	0.472
No. 11—Special silver-bearing, electrolytic	$\bar{X}$	33 420	33 570	33 080	33 440	32 750	32 840	32 900	39.7	39.2	40.7	37.8	37.8	37.2	42.3
	σ	304	261	117	156	367	51.9	123	2.88	0.625	1.89	1.31	1.31	3.80	0.944
No. 12—Special arsenical, electrolytic	$\bar{X}$	33 910	34 180	34 050	33 920	33 370	33 200	32 600	42.7	36.3	40.5	40.5	36.7	37.3	31.3
	σ	143	368	233	307	499	47.5	540	2.48	4.30	2.56	2.04	1.89	4.03	1.25

[a] Only two specimens tested.

(Continued on p. 630)

TABLE II.—*Continued.*

	Average ($\bar{X}$) and Standard Deviation (σ)	Tensile Strength, psi.							Elongation in 2 in., per cent						
		Original	1 yr.	2 yr.	3 yr.	5 yr.	8 yr.	10 yr.	Original	1 yr.	2 yr.	3 yr.	5 yr.	8 yr.	10 yr.
MARINE ATMOSPHERE															
No. 1—Electrolytic, N.E.C.	$\bar{X}$	33 760	34 100	33 490	33 910	33 100	33 720	33 050	42.1	45.7	43.2	42.2	45.5	41.0	40.3
	σ	141	141	353	112	103	220	248	2.40	5.33	3.06	1.23	1.08	2.12	0.624
No. 1A—Electrolytic, Chile	$\bar{X}$	32 060	32 300	31 680	32 100	31 530	32 020	31 700	42.8	40.3	41.3	41.3	39.3	40.2	40.8
	σ	248	268	194	140	124	203	308	0.898	0.624	0.624	0.471	1.21	3.41	3.12
No. 2—Prime Lake, Tamarack	$\bar{X}$	33 290	33 420	32 920	33 120	32 470	34 480	33 030	42.4	44.2	42.2	43.8	42.7	41.7	43.7
	σ	224	167	278	46.4	47.1	365	165	1.40	1.03	1.93	2.62	3.30	2.32	2.36
No. 3—Arsenical Lake, Tamarack	$\bar{X}$	35 580	35 480	35 020	35 100	34 480	34 860	34 530	44.0	44.0	44.0	42.5	44.2	44.2	42.2
	σ	369	197	49.7	250	246	213	125	1.78	2.87	0	1.78	0.624	2.66	1.28
No. 4—Arsenical Lake, Superior	$\bar{X}$	35 160	...	34 360	...	34 480	...	33 730	42.0	...	43.0	...	40.0	...	43.5
	σ	201	...	269	...	225	...	85.0	1.89	...	0.816	...	0.957	...	2.27
No. 5—Silver-bearing Lake, Franklin	$\bar{X}$	33 440	33 770	32 780	33 480	32 600	32 990	32 500	44.6	46.2	44.2	44.0	43.2	43.7	46.0
	σ	152	130	41.9	40.3	193	159	255	2.32	2.10	3.41	0	1.65	0.746	2.27
No. 8—Phosphorus-deoxidized, electrolytic	$\bar{X}$	34 250	34 320	33 730	34 210	33 580	33 990	33 400	40.8	39.8	37.0	38.2	37.8	40.2	38.8
	σ	307	147	440	0.974	548	85.0	634	1.94	2.90	1.68	1.55	1.65	0.624	2.25
No. 9—Fire-refined, Wallaroo	$\bar{X}$	35 390	35 490	35 210	35 620	34 900	35 300	34 530	44.2	42.7	44.3	42.3	44.8	42.3	44.0
	σ	330	108	32.7	43.2	294	81.7	103	2.53	1.93	2.06	1.03	2.25	0.471	0.707
No. 10—Fire-refined, A.B.S.	$\bar{X}$	32 640	32 530	32 150	32 540	32 030	31 750	31 470	40.2	42.3	40.2	41.2	41.8	41.8	40.3
	σ	213	340	626	244	206	350	262	1.07	3.40	0.850	1.55	1.89	2.72	1.31
No. 11—Special silver-bearing, electrolytic	$\bar{X}$	33 420	33 440	32 980	33 840	33 120	32 930	32 500	39.7	41.3	39.3	38.7	39.7	37.2	39.0
	σ	304	194	235	180	232	146	312	2.88	2.10	2.36	2.66	2.10	2.25	4.02
No. 12—Special arsenical, electrolytic	$\bar{X}$	33 910	34 750	33 060	34 260	33 770	33 580	33 230	42.7	37.7	37.0	37.7	37.8	37.5	38.3
	σ	143	171	840	478	170	618	943	2.48	2.72	3.19	0.624	1.03	4.27	1.93

RURAL ATMOSPHERE

No. 1—Electrolytic, N.E.C.	$\bar{X}$	33 760	34 170	33 510		33 370		33 650	42.1	45.5	43.8		40.7		42.7
	σ	141	114	143		249		141	2.40	2.49	1.84		0.85		0.624
No. 1A—Electrolytic, Chile	$\bar{X}$	32 060	32 340	31 730		31 850		31 720	42.8	42.5	40.7		45.0		41.8
	σ	248	26.2	336		334		225	0.898	3.51	1.03		0.817		1.84
No. 2—Prime Lake, Tamarack	$\bar{X}$	33 290	33 750	32 620		32 630		32 600	42.4	42.2	42.2		43.5		42.3
	σ	224	156	74.1		47.1		141	1.40	0.472	1.31		0.707		3.48
No. 3—Arsenical Lake, Tamarack	$\bar{X}$	35 580	35 640	35 140		35 120		34 880	44.0	43.8	45.2		44.2		43.7
	σ	369	139	479		306		125	1.78	0.624	2.46		1.31		1.18
No. 4—Arsenical Lake, Superior	$\bar{X}$	35 160	...	...		...		...	42.0	...	...		...		...
	σ	201	...	...		...		...	1.89	...	...		...		...
No. 5—Silver-bearing Lake, Franklin	$\bar{X}$	33 440	33 870	32 980		32 880		33 020	44.6	43.2	44.8		42.7		46.0
	σ	152	43.2	148		103		170	2.32	0.624	2.02		1.70		3.57
No. 8—Phosphorus-deoxidized, electrolytic	$\bar{X}$	34 250	34 240	34 540		34 050		34 030	40.8	42.0	39.8		39.7		40.2
	σ	307	398	374		324		188	1.94	2.55	1.31		1.55		1.43
No. 9—Fire-refined, Wallaroo	$\bar{X}$	35 390	35 630	35 510		34 750		35 220	44.2	43.0	42.0		46.3		42.8
	σ	330	186	716		227		47.1	2.53	1.64	3.54		2.01		1.25
No. 10—Fire-refined, A.B.S.	$\bar{X}$	32 640	33 130	31 620		31 800		32 100	40.2	40.0	44.2		40.2		42.0
	σ	213	391	843		356		0	1.07	1.42	4.37		2.96		1.47
No. 11—Special silver-bearing, electrolytic	$\bar{X}$	33 420	33 680	33 230		32 730		33 150	39.7	35.7	36.7		42.5		40.2
	σ	304	81.8	114		236		70.7	2.88	2.50	2.06		4.81		3.52
No. 12—Special arsenical, electrolytic	$\bar{X}$	33 910	34 360	34 110		34 080		34 130	42.7	37.0	37.5		42.7		41.3
	σ	143	163	139		165		206	2.48	1.08	1.78		1.18		0.472

(Concluded on p. 632)

TABLE II.—*Concluded.*
SEA WATER, HALF TIDE

	Average ($\bar{X}$) and Standard Deviation (σ)	Original	½ yr.	1 yr.	2 yr.	Original	½ yr.	1 yr.	2 yr.
No. 1—Electrolytic, N.E.C.	$\bar{X}$	33 760	32 150	32 290	30 970	42.1	45.2	43.7	40.2
	σ	141	187	305	166	2.40	2.02	2.02	3.52
No. 1A—Electrolytic, Chile	$\bar{X}$	32 060	30 880	30 390	29 720	42.8	41.5	41.3	37.3
	σ	248	239	118	248	0.898	1.08	1.25	0.850
No. 2—Prime Lake, Tamarack	$\bar{X}$	33 290	31 810	31 970	30 830	42.4	41.7	40.2	42.5
	σ	224	308	35.6	123	1.40	1.43	1.03	1.08
No. 3—Arsenical Lake, Tamarack	$\bar{X}$	35 580	34 520	34 830	33 720	44.0	43.8	43.0	40.3
	σ	369	184	104	289	1.78	0.624	2.68	0.945
No. 4—Arsenical Lake, Superior	$\bar{X}$	35 160	34 200	34 180	33 950	42.0	43.5	44.3	43.7
	σ	201	327	231	671	1.89	0.409	2.32	2.59
No. 5—Silver-bearing Lake, Franklin	$\bar{X}$	33 440	32 430	31 940	31 160	44.6	43.5	44.0	42.3
	σ	152	386	161	207	2.32	1.23	1.87	1.25
No. 8—Phosphorus-deoxidized, electrolytic	$\bar{X}$	34 250	32 410	32 620	31 860	40.8	37.8	37.2	36.5
	σ	307	317	252	331	1.94	4.03	3.57	1.08
No. 9—Fire-refined, Wallaroo	$\bar{X}$	35 390	34 480	34 120	33 130	44.2	41.7	40.8	39.8
	σ	330	47.1	223	54.4	2.53	1.55	0.236	1.31
No. 10—Fire-refined, A.B.S.	$\bar{X}$	32 640	31 320	30 890	29 680	40.2	40.3	38.8	42.5
	σ	213	225	442	61.9	1.07	3.70	1.70	2.12
No. 11—Special silver-bearing, electrolytic	$\bar{X}$	33 420	31 800	31 930	30 920	39.7	38.5	38.3	39.0
	σ	304	245	206	49.3	2.88	1.47	0.850	0.707
No. 12—Special arsenical, electrolytic	$\bar{X}$	33 910	34 020	33 170	32 760	42.7	38.5	40.2	36.3
	σ	143	131	525	333	2.48	2.16	1.65	3.68

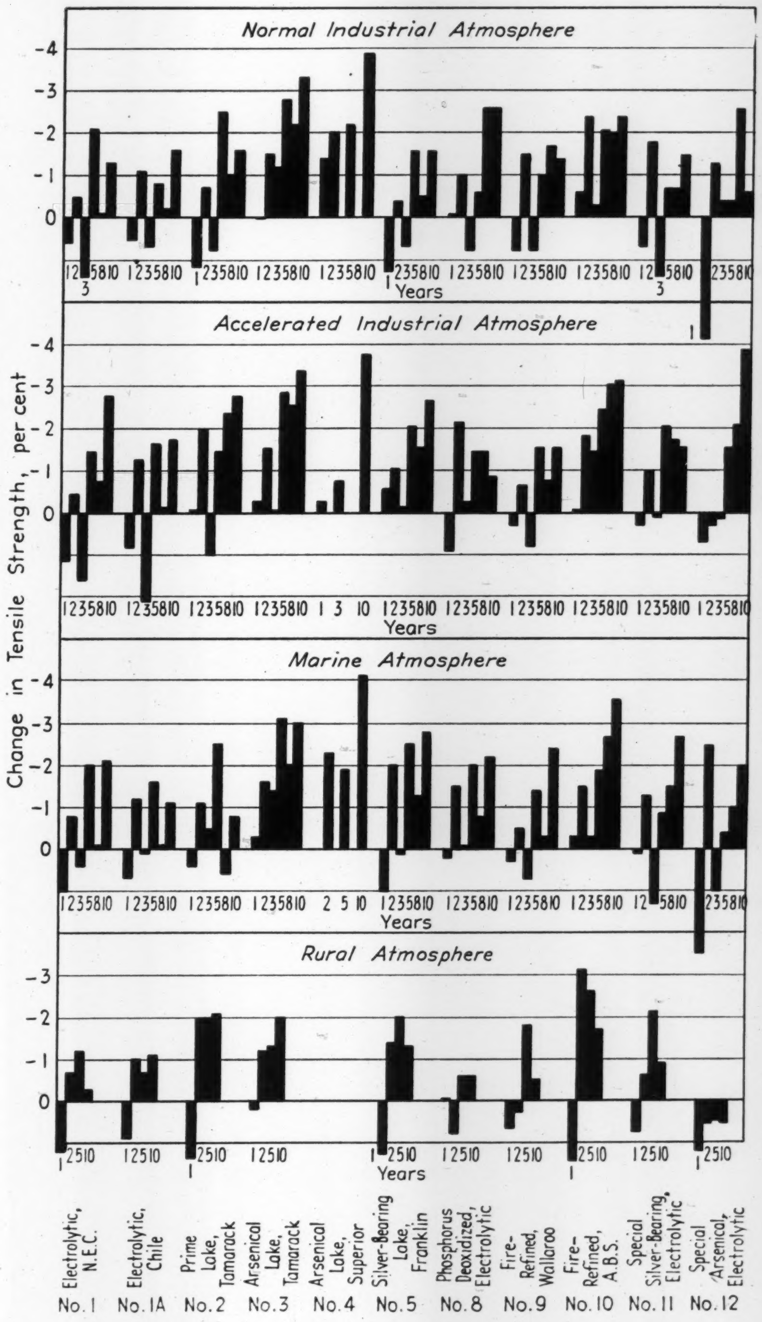


FIG. 11.—Effect of Various Location Atmospheres on Tensile Strength of Sheet Metal.

speckled with tiny black spots; all the other coppers were tarnished to a yellow-brown with scattered tiny black spots. At 2 yr., the relative difference between the two groups was about the same, but all were darker in color, and at 3 yr. the arsenical coppers were definitely black. At 5 yr. all specimens had begun to show a grayish overcast at scattered areas, but the arsenical coppers were patina-green on the edges. Specimens exposed 8 yr. had patina on the edges but the green color on the arsenical coppers had begun to creep down across the faces of the specimens. By the end of 10 yr., all specimens were well coated with patina, yet the arsenical coppers had a heavier patina.

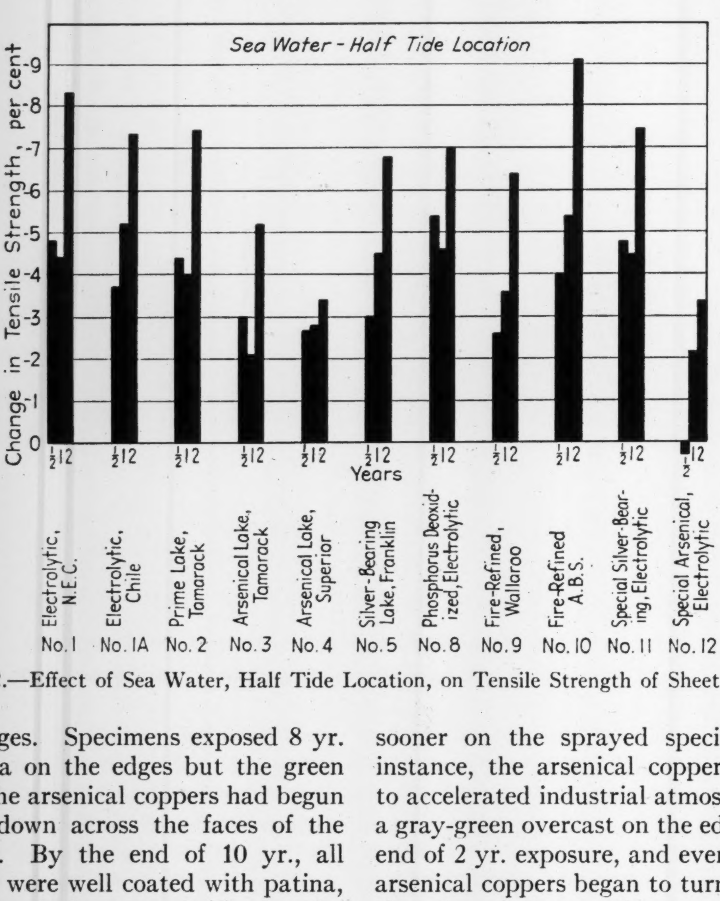
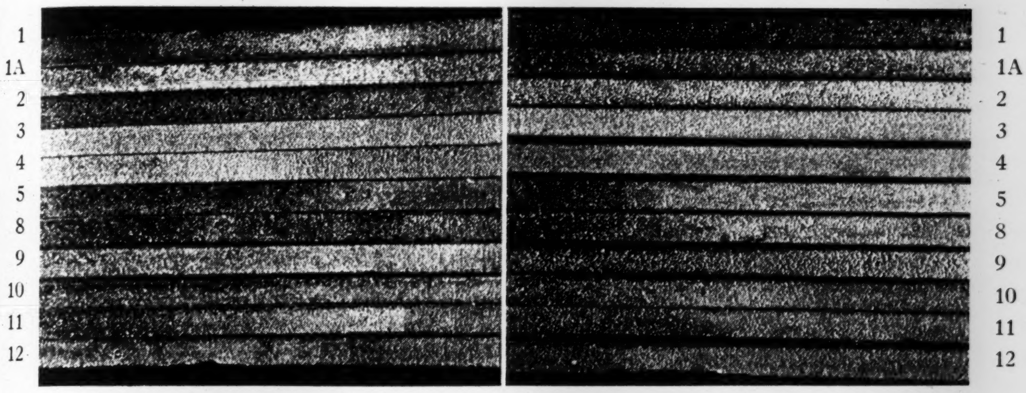


FIG. 12.—Effect of Sea Water, Half Tide Location, on Tensile Strength of Sheet Metal.

Figure 13 (*a*) shows the roughening and pitting on the top edges of specimens exposed 10 yr. There is no apparent difference in the amount of pitting among the specimens. In this and the following figures of these specimens, the severe indentations on the outer edges of samples Nos. 1 and 12 were caused by mechanical damage and should be neglected.

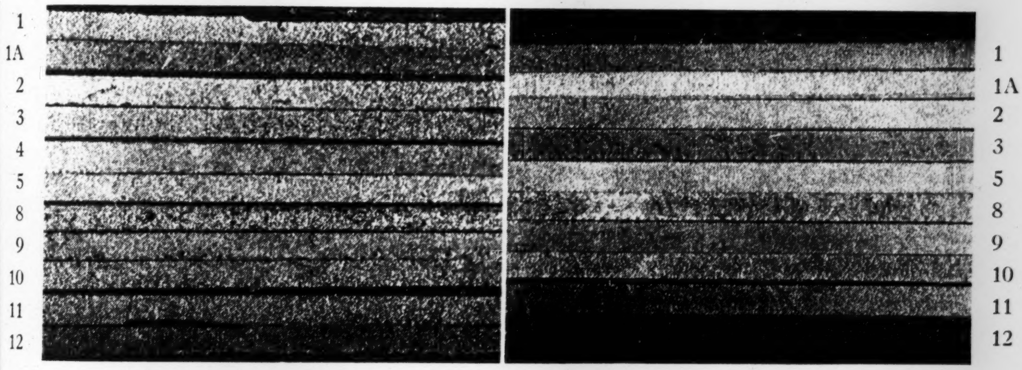
Accelerated Industrial Atmosphere.—The surface changes on the water-sprayed specimens were practically the same as those exposed to normal industrial atmosphere, but changes appeared sooner on the sprayed specimens; for instance, the arsenical coppers exposed to accelerated industrial atmosphere had a gray-green overcast on the edges at the end of 2 yr. exposure, and even the non-arsenical coppers began to turn green on the edges at 3 yr. After 8 yr. all specimens were well coated with patina.

Figure 13 (*b*) shows the roughness and pitting on the top edges of specimens exposed to accelerated atmospheric corrosion for 10 yr. The arsenical lake



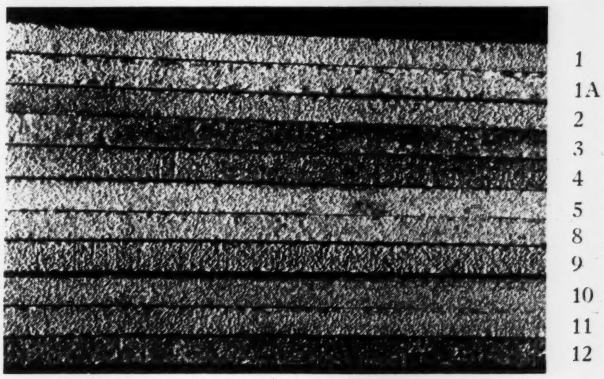
(*a*) Normal industrial atmosphere, exposed 10 yr.

(*b*) Accelerated industrial atmosphere, exposed 10 yr.



(*c*) Marine atmosphere, exposed 10 yr.

(*d*) Rural atmosphere, exposed 10 yr.



(*e*) Sea water, half tide location, exposed 2 yr.

No. 1	Electrolytic, N.E.C.
No. 1A	Electrolytic, Chile.
No. 2	Prime Lake, Tamarack.
No. 3	Arsenical Lake, Tamarack.
No. 4	Arsenical Lake, Superior.
No. 5	Silver-bearing Lake, Franklin.
No. 8	Phosphorus-deoxidized, electrolytic.
No. 9	Fire-refined, Wallaroo.
No. 10	Fire-refined, A.B.S.
No. 11	Special silver-bearing, electrolytic.
No. 12	Special arsenical, electrolytic.

FIG. 13.—Condition of Top Edge of Sheet Metal Specimens (× 4).

TABLE III.—WIRE SPECIMENS.

	Average ($\bar{X}$) and Standard Deviation (σ)	Breaking Load, lb.							Elongation in 10 in., per cent						
		Original	1 yr.	2 yr.	3 yr.	5½ yr.	8½ yr.	10½ yr.	Original	1 yr.	2 yr.	3 yr.	5½ yr.	8½ yr.	10½ yr.
NORMAL INDUSTRIAL ATMOSPHERE															
No. 1—Electrolytic, N.E.C.	$\bar{X}$	824.0	806.0	789.0	802.7	805.0	782.0	809.7	1.31	1.33	0.98	1.09	1.23	1.15	1.39
	σ	2.45	14.6	1.63	3.09	6.70	8.29	6.00	0.0283	0.148	0.0499	0.0206	0.128	0.0497	0.192
No. 1A—Electrolytic, Chile	$\bar{X}$	815.3	828.7	817.3	812.3	806.3	810.7	824.4	1.03	1.17	1.14	0.84	1.07	0.92	0.85
	σ	12.3	4.71	19.4	21.7	23.0	9.68	9.71	0.204	0.0287	0.0918	0.202	0.187	0.0616	0.0928
No. 2—Prime Lake, Tamarack	$\bar{X}$	825.3	809.7	819.0	820.7	816.3	811.7	819.5	1.23	1.10	0.96	1.02	1.07	0.93	1.25
	σ	0.479	9.68	7.36	4.50	4.50	1.25	8.87	0.0804	0.0910	0.134	0.0330	0.0497	0.0141	0.0624
No. 3—Arsenical Lake, Tamarack	$\bar{X}$	909.7	928.3	917.3	929.7	919.0	908.7	916.5	1.04	1.05	0.78	0.91	1.02	0.74	1.03
	σ	14.8	0.471	15.9	9.28	23.0	3.40	4.21	0.0294	0.0566	0.0478	0.0532	0.116	0.0544	0.158
No. 4—Arsenical Lake, Superior	$\bar{X}$	901.3	897.0	900.7	906.0	894.3	824.0	891.9	0.92	0.91	0.63	0.84	1.01	0.65	0.87
	σ	2.06	3.40	1.70	1.64	4.50	73.6	20.8	0.0579	0.0741	0.0216	0.0249	0.0660	0.0300	0.0572
No. 5—Silver-bearing Lake, Franklin	$\bar{X}$	844.7	836.3	839.3	832.3	829.0	817.3	830.8	1.15	1.20	0.92	0.97	1.17	1.00	1.22
	σ	5.56	3.30	2.88	10.4	17.7	20.2	13.2	0.0910	0.0655	0.0170	0.0544	0.140	0.0544	0.0816
No. 8—Phosphorus-deoxidized, electrolytic	$\bar{X}$	891.3	884.7	891.3	896.7	884.0	873.0	887.6	1.45	1.52	1.13	1.19	1.42	1.14	1.41
	σ	7.78	2.06	4.79	7.88	8.49	7.87	5.23	0.203	0.0125	0.0536	0.0163	0.0707	0.0206	0.0141
No. 9—Fire-refined, Wallaroo	$\bar{X}$	849.0	841.3	849.7	858.7	842.0	835.7	850.6	1.12	1.17	1.02	0.97	1.02	1.03	1.29
	σ	3.74	7.41	1.70	1.89	3.27	2.06	5.55	0.151	0.120	0.231	0.0419	0.0356	0.0249	0.106
No. 10—Fire-refined, A.B.S.	$\bar{X}$	827.7	831.3	832.0	840.0	831.3	817.0	833.1	1.21	1.21	0.93	1.11	1.18	0.85	1.44
	σ	0.471	9.54	4.32	9.80	0.943	10.2	4.70	0.0817	0.0990	0.0707	0.0789	0.123	0.0858	0.0779
No. 11—Special silver-bearing, electrolytic	$\bar{X}$	840.0	824.7	840.3	845.3	845.7	815.0	837.3	1.26	1.24	1.02	1.12	1.16	0.95	1.37
	σ	2.16	18.5	4.03	3.78	10.7	12.1	3.35	0.0125	0.0754	0.0497	0.0490	0.0532	0.0141	0.101
No. 12—Special arsenical, electrolytic	$\bar{X}$	906.3	905.0	901.3	901.3	899.0	878.3	901.5	1.06	1.29	0.91	0.99	1.15	0.94	1.20
	σ	1.70	2.38	3.70	15.9	1.41	10.5	0.818	0.0536	0.114	0.105	0.119	0.0411	0.0990	0.0974

ACCELERATED INDUSTRIAL ATMOSPHERE

No. 1—Electrolytic, N.E.C.	$\bar{X}$	824.0	806.3	827.3	816.3	793.3	807.3	816.6	1.31	1.15	1.05	1.18	1.21	1.08	1.23
	σ	2.45	6.95	0.832	6.13	19.1	2.36	7.35	0.0283	0.0464	0.0294	0.0772	0.0899	0.0249	0.136
No. 1A—Electrolytic, Chile	$\bar{X}$	815.3	777.7	833.7	806.3	810.7	812.7	810.3	1.03	1.23	0.82	1.00	0.86	0.95	1.13
	σ	12.3	46.7	20.5	21.0	6.19	6.85	41.5	0.204	0.383	0.172	0.0956	0.210	0.0665	0.249
No. 2—Prime Lake, Tamarack	$\bar{X}$	825.3	818.3	810.0	824.0	819.3	807.3	815.4	1.23	1.10	0.86	0.95	1.06	0.93	1.21
	σ	0.479	7.85	22.7	1.63	4.19	1.70	6.14	0.0804	0.0519	0.0432	0.0206	0.0990	0.0163	0.0818
No. 3—Arsenical Lake, Tamarack	$\bar{X}$	909.7	884.7	928.0	921.7	906.0	895.7	908.9	1.04	0.94	0.84	0.83	0.98	0.73	1.13
	σ	14.8	32.2	6.40	5.91	21.6	1.25	6.59	0.0294	0.0988	0.0432	0.0490	0.0556	0.0419	0.0728
No. 4—Arsenical Lake, Superior	$\bar{X}$	901.3	900.0	899.0	905.0	896.0	878.3	893.7	0.92	0.84	0.74	0.77	0.86	0.68	1.06
	σ	2.06	6.11	10.7	3.56	4.46	1.25	1.95	0.0579	0.116	0.0616	0.0141	0.184	0.0283	0.0464
No. 5—Silver-bearing Lake, Franklin	$\bar{X}$	844.7	...	841.3	834.7	838.7	804.7	840.7	1.15	...	0.87	1.03	1.11	0.95	1.23
	σ	5.56	...	3.86	16.4	1.89	12.5	3.30	0.0910	...	0.123	0.0236	0.0822	0.0411	0.0411
No. 8—Phosphorus-deoxidized, electrolytic	$\bar{X}$	891.3	890.5[a]	880.3	887.7	870.7	865.3	877.4	1.45	1.45	1.22	1.31	1.46	1.21	1.44
	σ	7.78	0.435	16.7	7.72	2.50	6.95	16.2	0.0203	0.111	0.0920	0.0660	0.0759	0.0829	0.0424
No. 9—Fire-refined, Wallaroo	$\bar{X}$	849.0	849.7	847.0	852.3	844.7	830.7	849.3	1.12	1.26	0.88	1.00	1.27	0.96	1.24
	σ	3.74	2.05	2.83	0.471	2.63	0.943	6.61	0.0817	0.0499	0.0665	0.0455	0.0287	0.0245	0.00943
No. 10—Fire-refined, A.B.S.	$\bar{X}$	827.7	821.3	836.7	839.7	821.0	818.0	827.3	1.21	1.33	0.95	1.01	1.29	1.02	1.27
	σ	0.471	2.49	12.0	4.19	7.88	7.49	1.89	0.0817	0.169	0.0881	0.0497	0.161	0.0374	0.0262
No. 11—Special silver-bearing, electrolytic	$\bar{X}$	840.0	836.7	851.3	845.7	840.0	825.7	835.0	1.26	1.26	1.09	1.09	1.27	1.19	1.31
	σ	2.16	0.471	7.41	4.19	2.93	3.40	1.16	0.0125	0.0653	0.0525	0.0510	0.0450	0.0283	0.0736
No. 12—Special arsenical, electrolytic	$\bar{X}$	906.3	904.7	910.3	911.7	890.3	871.0	902.2	1.06	1.06	0.93	0.99	1.19	0.93	1.22
	σ	1.70	2.49	3.30	3.86	12.5	17.6	1.92	0.0536	0.0189	0.0125	0	0.00943	0.0262	0.0170

[a] Six specimens tested

(Concluded on p. 638)

TABLE III. *Continued.*

	Average ($\bar{X}$) and Standard Deviation (σ)	Breaking Load, lb.							Elongation in 10 in., per cent						
		Original	1 yr.	2 yr.	3 yr.	5½ yr.	8½ yr.	10½ yr.	Original	1 yr.	2 yr.	3 yr.	5½ yr.	8½ yr.	10½ yr.
MARINE ATMOSPHERE															
No. 1—Electrolytic, N.E.C.	$\bar{X}$	824.0	811.7	826.0	812.7	815.7	796.3	816.5	1.31	1.28	1.04	1.09	1.09	1.08	1.13
	σ	2.45	3.69	5.98	22.5	14.6	4.50	1.64	0.0283	0.129	0	0.00471	0.0493	0.0125	0.0556
No. 1A—Electrolytic, Chile	$\bar{X}$	815.3	801.0	839.7	828.3	798.3	816.0	...	1.03	0.88	1.01	0.90	0.83	0.96	1.10
	σ	12.3	17.0	20.5	4.19	2.63	2.06	...	0.204	0.123	0.110	0.104	0.156	0.0424	0.0536
No. 2—Prime Lake, Tamarack	$\bar{X}$	825.3	824.0	832.3	833.7	791.7	810.0	821.0	1.23	1.19	0.99	1.01	0.92	0.99	1.23
	σ	0.479	2.93	8.19	5.32	42.9	2.06	7.30	0.0804	0.0698	0.0668	0.0544	0.110	0.0189	0.0704
No. 3—Arsenical Lake, Tamarack	$\bar{X}$	909.7	923.0	934.3	931.0	916.0	906.3	926.5	1.04	0.96	0.81	0.86	0.93	0.86	1.14
	σ	14.8	3.56	12.7	6.48	11.8	3.86	2.86	0.0294	0.0262	0.137	0.0660	0.0294	0.0419	0.235
No. 4—Arsenical Lake, Superior	$\bar{X}$	901.3	899.3	913.7	906.7	899.7	889.7	894.0	0.92	0.98	0.74	0.86	0.78	0.86	0.80
	σ	2.06	4.11	1.70	6.19	0.473	1.25	12.3	0.0579	0.0634	0.0141	0.0294	0.0249	0.0283	0.225
No. 5—Silver-bearing Lake, Franklin	$\bar{X}$	844.7	837.3	849.3	847.7	837.0	820.7	832.3	1.15	1.19	0.86	1.02	0.96	1.03	1.44
	σ	5.56	2.50	0.471	2.06	4.25	6.19	19.5	0.0910	0.0899	0.0579	0.0121	0.0525	0.0249	0.0286
No. 8—Phosphorus-deoxidized, electrolytic	$\bar{X}$	891.3	887.3	889.0	881.0	890.0	870.0	886.9	1.45	1.46	1.31	1.35	1.46	1.31	1.44
	σ	7.78	4.50	7.07	5.36	5.72	4.97	6.16	0.203	0.138	0.0838	0.0377	0.0532	0.0829	0.0624
No. 9—Fire-refined, Wallaroo	$\bar{X}$	849.0	850.0	861.7	857.7	842.3	837.7	854.4	1.12	1.19	0.98	1.10	1.08	1.09	1.25
	σ	3.74	2.16	0.943	0.471	1.25	1.70	0.849	0.151	0.134	0.0356	0.0170	0.0455	0.120	0.0748
No. 10—Fire-refined, A.B.S.	$\bar{X}$	827.7	828.0	841.7	836.7	824.7	821.3	836.5	1.21	1.43	1.09	1.10	0.99	1.17	1.38
	σ	0.471	2.93	8.34	6.59	12.2	2.63	4.37	0.0817	0.178	0.0741	0.135	0.0141	0.0573	0.0704
No. 11—Special silver-bearing, electrolytic	$\bar{X}$	840.0	839.0	847.3	836.7	826.3	825.0	845.8	1.26	1.26	1.00	1.18	1.28	1.17	1.42
	σ	2.16	1.41	8.96	12.5	15.1	0.817	4.25	0.0125	0.00817	0.0694	0.0464	0.108	0.0544	0.0163
No. 12—Special arsenical, electrolytic	$\bar{X}$	906.3	902.7	916.3	897.3	903.7	887.0	903.0	1.06	1.17	0.89	1.06	1.03	1.06	1.11
	σ	1.70	3.09	7.07	10.9	3.30	4.97	2.74	0.0536	0.0883	0.0283	0.105	0.114	0.0245	0.203

RURAL ATMOSPHERE

No. 1—Electrolytic, N.E.C.	$\bar{X}$	824.0	798.7	829.7	827.0	813.3	815.0	859.3	1.31	1.24	1.07	1.09	1.26	1.04	1.23
	σ	2.45	1.25	6.34	7.79	17.2	1.41	21.6	0.0283	0.160	0.0283	0.0665	0.0967	0.00471	0.0819
No. 1A—Electrolytic, Chile	$\bar{X}$	815.3	791.7	836.3	831.7	806.3	808.7	839.9	1.03	1.28	0.91	0.90	0.84	0.99	1.19
	σ	12.3	57.8	15.8	22.4	8.99	30.2	36.5	0.204	0.109	0.170	0.183	0.303	0.0900	0.0713
No. 2—Prime Lake, Tamarack	$\bar{X}$	825.3	820.7	840.7	829.7	826.0	813.7	827.5	1.23	1.15	1.05	1.02	0.95	0.94	1.30
	σ	0.479	0.943	5.73	4.50	9.80	8.99	4.72	0.0804	0.0822	0.0330	0.0287	0.0309	0.0330	0.0519
No. 3—Arsenical Lake, Tamarack	$\bar{X}$	909.7	920.7	945.7	923.7	926.7	917.3	934.3	1.04	1.02	0.83	0.95	0.92	0.86	1.09
	σ	14.8	2.62	4.03	11.3	7.72	3.40	0.998	0.0294	0.00471	0.0865	0.00817	0.0386	0.0519	0.0419
No. 4—Arsenical Lake, Superior	$\bar{X}$	901.3	899.3	923.0	908.0	906.0	896.0	911.9	0.92	0.93	0.82	0.86	0.84	0.78	0.98
	σ	2.06	3.40	2.45	5.65	2.06	1.41	2.45	0.0579	0.0246	0.0403	0.0732	0.0638	0.0189	0.106
No. 5—Silver-bearing Lake, Franklin	$\bar{X}$	844.7	840.7	858.0	844.7	846.0	820.7	848.9	1.15	1.18	0.91	1.03	1.08	0.88	1.35
	σ	5.56	1.25	5.72	6.24	2.94	19.3	0.589	0.0910	0.0262	0.0881	0.0206	0.0249	0.0685	0.144
No. 8—Phosphorus-deoxidized, electrolytic	$\bar{X}$	891.3	877.0	893.7	888.3	892.0	872.0	892.2	1.45	1.43	1.31	1.30	1.38	1.22	1.53
	σ	7.78	6.38	2.36	6.02	5.25	4.32	1.27	0.203	0.145	0.0536	0.0704	0.00471	0.0340	0.157
No. 9—Fire-refined, Wallaroo	$\bar{X}$	849.0	844.7	863.7	848.7	848.0	839.7	856.3	1.12	1.30	1.07	1.12	1.28	1.04	1.31
	σ	3.74	4.50	6.34	4.71	6.38	3.30	6.33	0.151	0.0860	0.0450	0.0519	0.152	0.0838	0.102
No. 10—Fire-refined, A.B.S.	$\bar{X}$	827.7	819.0	840.3	834.7	829.0	821.0	855.9	1.21	1.35	1.03	1.09	1.18	1.01	1.40
	σ	0.471	4.97	13.1	5.73	6.98	3.74	26.4	0.0817	0.0896	0.0340	0.0510	0.141	0.0849	0.0490
No. 11—Special silver-bearing, electrolytic	$\bar{X}$	840.0	827.0	841.7	844.3	839.3	835.0	839.4	1.26	1.31	1.05	1.14	1.22	1.08	1.19
	σ	2.16	14.9	11.2	3.86	2.62	4.24	9.13	0.0125	0.0532	0.00943	0.0613	0.0478	0.0831	0.0525
No. 12—Special arsenical, electrolytic	$\bar{X}$	906.3	893.0	923.7	909.3	904.7	897.3	897.8	1.06	1.02	0.94	1.08	1.15	0.98	1.31
	σ	1.70	8.29	2.06	0.943	5.19	3.30	13.2	0.0536	0.0262	0.0455	0.0249	0.0249	0.0519	0.0125

coppers, Nos. 3 and 4, appear to be corroded somewhat more uniformly than the average, while deoxidized copper is the roughest.

Marine Atmosphere.—All specimens at the marine atmosphere exposure acquired a green overcast during the first year, but the undercoat on the arsenical coppers was darker than on the other coppers. The patina was not complete on any of the specimens, even at 10 yr.

Figure 13 (*c*) shows that the specimens exposed to marine atmosphere were but slightly roughened. Deoxidized copper, No. 8, is roughened more than the others.

Rural Atmosphere.—The specimens exposed to rural atmosphere had a green overcast at the end of the first year, as at the marine exposure station. The undercoat was brick-red except on the arsenical copper specimens, where it was dark brown. At 10 yr., the patina was almost uniform on all specimens.

Figure 13 (*d*) shows that only deoxidized copper, No. 8, is more roughened by corrosion than the others.

Sea Water—Half Tide Location:

At 6 months all specimens were coated with a fairly adherent, mossy-green scale.

At 2 yr. it was noted that the corrosion products on the arsenical coppers, Nos. 3, 4, and 12, and special silver-bearing electrolytic copper, No. 11, were not as adherent as on the other metals. The condition of the samples of each type of copper exposed 2 yr. after the corrosion products had been removed is given below:

No. 1. Electrolytic Copper, N.E.C.: Pitted at support holes, surface rough, edges deeply corroded.

No. 1A. Electrolytic Copper, Chile: Pitted at support holes, surface rough, edges deeply corroded.

No. 2. Prime Lake Copper, Tamarack: Pitted at support holes, surface rough, edges deeply corroded.

No. 3. Arsenical Lake Copper, Tamarack: Pits scattered all over surface, edges deeply corroded.

No. 4. Arsenical Lake Copper, Superior: Pitted at support holes, faces of specimens quite rough, edges still fairly sharp.

No. 5. Silver-Bearing Lake Copper, Franklin: Pitted at support holes, faces of specimens quite rough, edges fairly sharp.

No. 8. Phosphorus-Deoxidized Electrolytic Copper: Slight pitting at support holes, faces of specimens etched, edges corroded.

No. 9. Fire-Refined, Wallaroo Copper: Pitted over whole surface, edges corroded.

No. 10. Fire-Refined, A.B.S. Copper: Surfaces etched, edges fairly sharp.

No. 11. Special Silver-Bearing Electrolytic Copper: Pitted at support holes, surfaces etched, edges fairly sharp.

No. 12. Special Arsenical Electrolytic Copper: Tiny pits scattered over entire surface, edges corroded.

The condition of the top edges of specimens exposed to sea water at half tide location for 2 yr. may be seen in Fig. 13*e*.

WIRE SPECIMENS

Tension, elongation, and electrical resistance tests were made on the wire specimens to determine the rate of corrosion at the several test sites. The averages and standard deviations of the breaking loads in pounds are given in Table III. An examination of the data fails to indicate any definitely significant changes in tensile strength that can be attributed to corrosion.

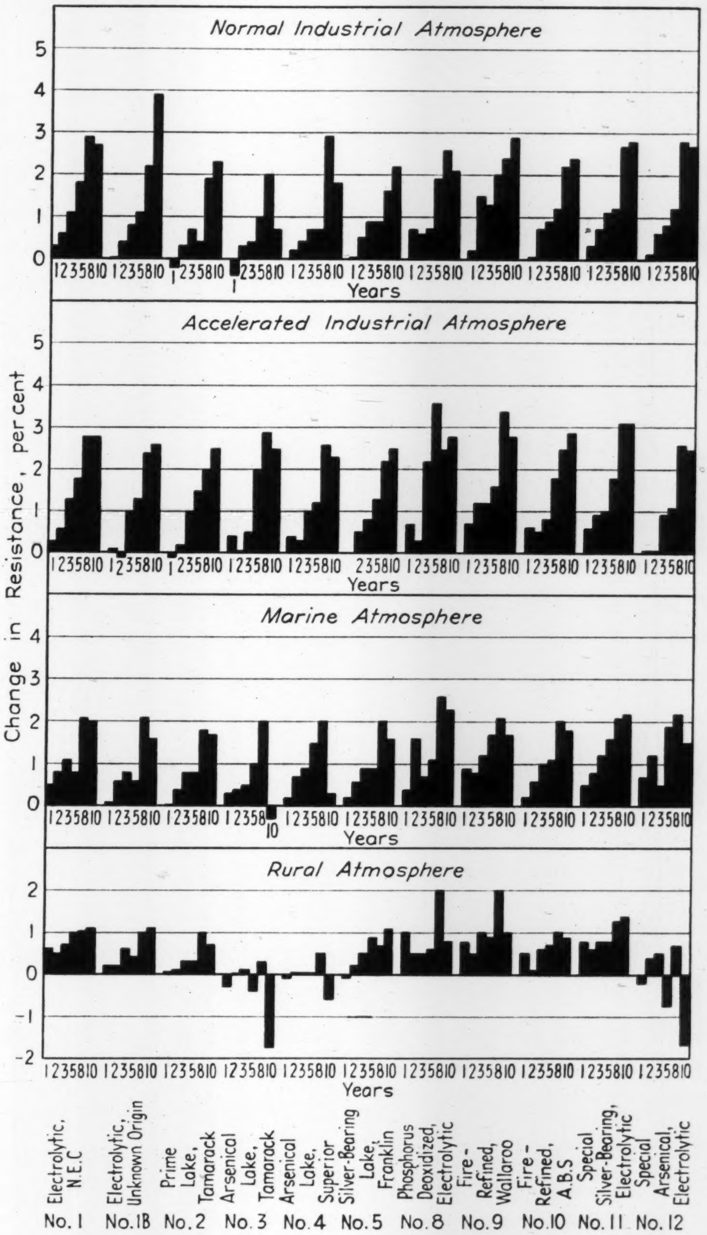


FIG. 14.—Effect of Various Location Atmospheres on Electrical Resistance of Wire Specimens.

TABLE IV.—WIRE SPECIMENS—ELECTRICAL RESISTANCE, MICROHMS PER FOOT AT 20 C.

	Average ($\bar{X}$) and Standard Deviation (σ)	Initial	1 yr.	2 yr.[a]	3 yr.	5½ yr.	8½ yr.	10½ yr.
NORMAL INDUSTRIAL ATMOSPHERE								
No. 1—Electrolytic, N.E.C.	$\bar{X}$	635.1	636.8	638.8	641.9	646.9	653.4	652.2
	σ	0.0817	1.02		1.28	1.14	0.525	1.65
No. 1A—Electrolytic, Chile	$\bar{X}$	640.3	640.5	642.6	645.3	647.6	654.1	655.4
	σ	0.525	0.455		0.910	0.772	1.89	0.829
No. 2—Prime Lake, Tamarack	$\bar{X}$	638.2	637.1	640.0	642.6	640.6	650.5	652.8
	σ	0.309	0.525		0.386	3.21	1.00	0.864
No. 3—Arsenical Lake, Tamarack	$\bar{X}$	1759.0	1752.0	1763.0	1766.0	1776.3	1794.7	1771.0
	σ	0.732	2.94		4.97	3.30	1.25	6.16
No. 4—Arsenical Lake, Superior	$\bar{X}$	1172.0	1174.0	1177.0	1180.0	1180.0	1206.3	1192.7
	σ	3.09	0.236		6.02	6.98	3.40	3.30
No. 5—Silver-bearing Lake, Franklin	$\bar{X}$	645.9	646.0	648.9	651.4	651.9	653.4	660.0
	σ	0.206	0.340		0.579	0.992	5.27	1.36
No. 8—Phosphorus deoxidized, electrolytic	$\bar{X}$	795.7	801.9	800.5	801.2	811.0	816.0	812.4
	σ	5.90	5.22		0.829	3.73	6.08	3.88
No. 9—Fire-refined, Wallaroo	$\bar{X}$	702.8	704.3	713.6	712.3	716.6	719.8	722.9
	σ	2.25	2.21		2.14	0.525	2.00	2.74
No. 10—Fire-refined, A.B.S.	$\bar{X}$	676.5	676.5	681.0	682.7	684.3	691.7	692.5
	σ	0.340	1.64		0.680	0.909	1.61	0.694
No. 11—Special silver-bearing, electrolytic	$\bar{X}$	638.9	640.6	643.1	646.0	646.7	655.9	656.5
	σ	0.411	0.0471		0.327	1.48	0.283	0.544
No. 12—Special arsenical, electrolytic	$\bar{X}$	1302.0	1303.0	1310.0	1312.0	1317.3	1339.3	1341.3
	σ	0.294	6.94		4.50	3.77	7.59	3.30
ACCELERATED INDUSTRIAL ATMOSPHERE								
No. 1—Electrolytic, N.E.C.	$\bar{X}$	635.1	637.2	639.1	643.7	646.3	652.8	652.7
	σ	0.0817	0.602		0.602	0.170	0.471	0.831
No. 1A—Electrolytic, Chile	$\bar{X}$	640.3	641.2	639.9	646.5	648.4	655.9	656.7
	σ	0.525	0.141		1.68	0.994	1.98	0.490
No. 2—Prime Lake, Tamarack	$\bar{X}$	638.2	637.3	639.7	644.3	647.6	650.8	654.2
	σ	0.309	0.236		0.125	1.04	0.910	0.748
No. 3—Arsenical Lake, Tamarack	$\bar{X}$	1759.0	1766.0	1759.0	1768.0	1794.3	1810.3	1803.0
	σ	0.732	5.56		6.02	15.6	2.06	4.97
No. 4—Arsenical Lake, Superior	$\bar{X}$	1172.0	1177.0	1175.0	1184.0	1186.3	1203.0	1199.3
	σ	3.09	2.94		4.03	4.11	2.16	3.68
No. 5—Silver-bearing Lake, Franklin	$\bar{X}$	645.9	......	649.3	651.0	654.2	660.0	662.3
	σ	0.206			1.58	0.850	0.188	0.655
No. 8—Phosphorus deoxidized, electrolytic	$\bar{X}$	795.7	801.5[b]	798.3	813.4	824.2	815.7	818.2
	σ	5.90	5.46		5.09	1.77	5.81	4.35
No. 9—Fire-refined, Wallaroo	$\bar{X}$	702.8	708.5	711.6	711.2	714.4	726.7	722.6
	σ	2.25	2.70		3.38	1.47	0	1.37
No. 10—Fire-refined, A.B.S.	$\bar{X}$	676.5	680.3	680.0	682.1	688.7	693.1	696.0
	σ	0.340	0.163		1.15	1.23	1.41	0.499
No. 11—Special silver-bearing, electrolytic	$\bar{X}$	638.9	642.6	644.4	645.3	650.5	658.4	658.9
	σ	0.411	0.0817		0.125	0.638	2.05	2.64
No. 12—Special arsenical, electrolytic	$\bar{X}$	1302.0	1302.0	1302.0	1314.0	1315.7	1336.3	1330.0
	σ	0.294	0.0471		0.817	13.9	9.53	4.32

[a] Only one specimen tested.
[b] Six specimens tested.

TABLE IV.—*Concluded.*

	Average ($\bar{X}$) and Standard Deviation (σ)	Initial	1 yr.	2 yr.[a]	3 yr.	5½ yr.	8½ yr.	10½ yr.
MARINE ATMOSPHERE								
No. 1—Electrolytic, N.E.C.	$\bar{X}$	635.1	638.4	640.2	642.1	639.6	648.4	647.7
	σ	0.0817	0.478		0.838	0.910	0.785	0.898
No. 1A—Electrolytic, Chile	$\bar{X}$	640.3	641.1	644.0	645.1	644.0	653.7	650.7
	σ	0.525	0.0471		0.136	0.772	0.736	0.283
No. 2—Prime Lake, Tamarack	$\bar{X}$	638.2	638.3	640.9	643.2	643.1	649.5	649.3
	σ	0.309	0		0.0473	0.579	0.377	0.579
No. 3—Arsenical Lake, Tamarack	$\bar{X}$	1759.0	1764.0	1766.0	1768.0	1776.7	1794.7	1754.0
	σ	0.732	3.86		4.50	6.24	1.70	2.83
No. 4—Arsenical Lake, Superior	$\bar{X}$	1172.0	1174.0	1180.0	1182.0	1190.3	1195.7	1175.7
	σ	3.09	3.30		2.06	7.41	3.09	2.50
No 5—Silver-bearing Lake, Franklin	$\bar{X}$	645.9	647.2	649.5	651.5	652.0	658.7	656.4
	σ	0.206	0.386		0.374	1.63	0.990	2.32
No. 8—Phospohrus deoxidized, electrolytic	$\bar{X}$	795.7	798.8	808.2	800.9	804.8	816.0	814.4
	σ	5.90	2.49		0.497	2.25	8.78	5.85
No. 9—Fire-refined, Wallaroo	$\bar{X}$	702.8	708.8	708.4	711.1	714.4	717.5	714.6
	σ	2.25	6.68		1.42	2.48	3.06	2.70
No. 10—Fire-refined, A.B.S.	$\bar{X}$	676.5	678.0	680.4	683.3	684.1	690.0	687.5
	σ	0.340	0.125		1.35	1.51	0.902	2.16
No. 11—Special silver-bearing, electrolytic	$\bar{X}$	638.9	642.1	644.3	646.8	649.4	652.7	653.1
	σ	0.411	0.236		0.826	0.579	1.23	1.57
No. 12—Special arsenical, electrolytic	$\bar{X}$	1302.0	1311.0	1318.0	1308.0	1326.7	1329.7	1322.0
	σ	0.294	2.06		8.19	6.19	5.32	2.83
RURAL ATMOSPHERE								
No. 1—Electrolytic, N.E.C.	$\bar{X}$	635.1	638.6	638.2	639.4	641.4	641.4	642.4
	σ	0.0817	0.600		0.712	0.432	0.713	0.287
No. 1A—Electrolytic, Chile	$\bar{X}$	640.3	641.8	641.9	643.9	643.0	646.8	647.1
	σ	0.525	0.206		0.432	8.17	0.170	0.736
No. 2—Prime Lake, Tamarack	$\bar{X}$	638.2	638.2	638.7	640.2	640.4	644.3	643.0
	σ	0.309	0.283		0.432	0.450	1.03	0.685
No. 3—Arsenical Lake, Tamarack	$\bar{X}$	1759.0	1754.0	1759.0	1761.0	1752.0	1765.0	1727.7
	σ	0.732	3.74		6.80	6.68	4.90	3.68
No. 4—Arsenical Lake, Superior	$\bar{X}$	1172.0	1171.0	1172.0	1172.0	1172.3	1178.3	1164.7
	σ	3.09	0.943		1.63	4.03	4.64	0.471
No. 5—Silver-bearing Lake, Franklin	$\bar{X}$	645.9	645.2	647.3	649.4	651.7	650.6	652.9
	σ	0.206	0.424		0.0471	2.36	1.75	0.735
No. 8—Phosphorus deoxidized, electrolytic	$\bar{X}$	795.7	804.0	799.7	799.5	800.8	811.7	801.7
	σ	5.90	9.70		4.68	7.19	9.02	5.14
No. 9—Fire-refined, Wallaroo	$\bar{X}$	702.8	708.8	706.1	709.9	708.8	716.6	709.8
	σ	2.25	1.70		3.33	1.63	0.712	2.68
No. 10—Fire-refined, A.B.S.	$\bar{X}$	676.5	680.0	677.0	680.4	681.0	683.8	682.8
	σ	0.340	0.125		0.829	0.817	1.49	0.963
No. 11—Special silver-bearing, electrolytic	$\bar{X}$	638.9	643.9	643.0	643.7	644.0	647.1	647.5
	σ	0.411	1.37		0.573	0.0817	0.831	2.10
No. 12—Special arsenical, electrolytic	$\bar{X}$	1302.0	1300.0	1307.0	1309.0	1291.3	1311.3	1280.0
	σ	0.294	6.85		4.50	7.43	1.70	5.65

The same conclusions are indicated from the data on elongation measurements also given in Table III. Some of the measurements on specimens exposed to normal and accelerated industrial weathering for $8\frac{1}{2}$ yr. had large losses in ductility, but due to large variations in elongation from specimen to specimen, they cannot be considered significant.

As previously mentioned, the wire specimens were exposed in the form of a bow so that raindrops or dewdrops would be retained at one area. Corrosion was not accelerated enough at the low point on the bow to cause a significant number of wires to break there in the tension tests.

An attempt has been made to determine the extent of corrosion by measuring the changes in electrical resistance of the wire specimens with time. Burns and Campbell,[6] and Hudson[7] have used similar methods and obtained good agreement with weight loss determinations.

In the tests reported here, resistance measurements were made in the laboratory on the same specimens used subsequently for tension tests. The average resistances in microhms per foot at 20 C. and the standard deviations are given in Table IV. Resistance measurements were made on only one specimen from each group of three wires exposed 2 yr., so no standard deviations are given for that period. A study of the changes in resistance and the standard deviations indicate that most wires were significantly corroded only after 5 yr. in the industrial exposures. Only those wires exposed to marine weathering for $8\frac{1}{2}$ yr. were corroded significantly as a group. Wires exposed to rural atmosphere were not all significantly corroded in any of the exposure periods. The percentage changes in resistance are shown graphically in Fig. 14.

It is not possible to determine statistically from the percentage changes in resistance whether one type of copper differs in corrosion rate from the average, as explained in connection with tension test data on sheet metal specimens. Therefore, it is necessary to depend entirely on judgment in evaluating the tests.

It is evident from the graph for percentage gain in resistance of the specimens exposed to normal industrial atmosphere that the corrosion rates for the electrolytic copper are somewhat above the average, and the corrosion rate for arsenical lake copper, No. 3, is somewhat below the average, but arsenical electrolytic copper, No. 12, is apparently not significantly better than the average. The corrosion rates of the various types of copper wire specimens determined by resistance measurements do not agree well with the corrosion rates of the sheet specimens as determined by weight measurements.

The amount of corrosion indicated by percentage gains in electrical resistance for the wires tested in accelerated industrial atmosphere, as represented by the bar charts in Fig. 14, does not show the same trends as in normal industrial atmosphere. It will be recalled that the penetrations in inches per year on the strip specimens were quite similar for each type of copper in the two atmospheres except, of course, that in all cases the rates were higher in the accelerated industrial weathering test.

It might be argued that the weight losses do not effectively show surface roughening and pitting, which might well be shown by changes in electrical resistance; yet, when these percentage changes in Fig. 14 are compared with the relative surface roughening determined by inspection under a 10-power hand glass as given in Table V, there is no relation between surface roughness and percentage gain in resistance of the 10-yr. specimens.

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[6] R. M. Burns and W. E. Campbell, "Electrical Resistance Method of Measuring Corrosion of Lead by Acid Vapors," *Transactions*, Am. Electrochemical Soc., Vol. 55, p. 271 (1929).

[7] J. C. Hudson, "Atmospheric Corrosion of Metals—Third Experimental Report to the Atmospheric Corrosion Research Committee," *Transactions*, Faraday Soc., Vol. 25, p. 177 (1929).

The general trends of corrosion of specimens at the marine location shown in Fig. 14 follow those in normal industrial atmosphere, although the percentage gains in resistance of the wires exposed to marine weathering are definitely lower than in industrial atmosphere. For some unknown reason, the apparent amount of corrosion of the 10-yr. specimens was less than the 8-yr. specimens. In general, this confirms the results of the tension tests for the 8- and 10-yr. groups. The corroded wires were all still quite smooth as were those exposed to rural atmosphere (to be discussed next), so no differences in resistance could be attributed to pitting or roughness.

The electrical resistance of wires exposed to the rural atmosphere indicates, as did the losses in weight on the sheet metal specimens, that the rural atmosphere was very mildly corrosive and that the arsenical coppers were more resistant to corrosion by this type of atmosphere than the other types of copper tested.

TABLE V.—SURFACE ROUGHENING OF WIRES EXPOSED 10 YR.

	Normal Industrial Atmosphere	Accelerated Industrial Atmosphere
No. 1—Electrolytic copper, N.E.C.	Roughened[a]	Roughened[a]
No. 1B—Electrolytic copper, unknown origin	Less than No. 1	Same as No. 1
No. 2—Prime Lake, Tamarack	Same as No. 1	Same as No. 1
No. 3—Arsenical Lake, Tamarack	More than No. 1	Less than No. 1
No. 4—Arsenical Lake, Superior	More than No. 1	Much more than No. 1
No. 5—Silver-bearing Lake, Franklin	Much more than No. 1	Less than No. 1
No. 8—Phosphorus-deoxidized, electrolytic	Same as No. 1	Same as No. 1
No. 9—Fire-refined, Wallaroo	Same as No. 1	Same as No. 1
No. 10—Fire-refined, A.B.S.	Same as No. 1	More than No. 1
No. 11—Special silver-bearing, electrolytic	More than No. 1	More than No. 1
No. 12—Special arsenical, electrolytic	Much more than No. 1	More than No. 1

[a] No. 1 arbitrarily chosen as basis for comparing the degree of roughening.

SUMMARY

Electrolytic copper, N.E.C. and Chile; prime lake copper, Tamarack; arsenical lake copper, Tamarack and Superior, and special arsenical electrolytic copper; silver-bearing lake copper, Franklin and special silver-bearing electrolytic copper, fire-refined Wallaroo and A.B.S. copper; and phosphorus-deoxidized electrolytic copper in the form of cold-rolled and annealed sheet metal 0.050 in. thick or in the form of hard-drawn wires $\frac{1}{8}$ in. in diameter or both have been exposed to normal industrial, accelerated industrial, marine, and rural atmospheres for several periods up to 10 yr. Sheet-metal specimens were also exposed to sea water corrosion at half tide for three periods up to 2 yr.

The amount of corrosion was determined on the sheet-metal specimens by changes in weight, tensile strength, and elongation. The amount of corrosion on wire specimens was determined by changes in tensile strength, elongation, and electrical resistance.

The significance of the weight change data has been determined by statistical analyses.

The measurement of changes in tensile

properties on sheet metal and wire specimens exposed to the several atmospheres was not delicate enough to detect differences in corrosion rates among the various types of copper.

The determination of changes in electrical conductivity of copper wire specimens to detect differences in amounts of corrosion gave results which were significant, yet the agreement with loss in weight determinations was poor in many cases.

The weight change data indicate that in industrial and marine atmospheres high-purity electrolytic copper and fire-refined Wallaroo copper (the latter with 0.25 per cent nickel) are more corrosion resistant than the general average and the arsenical lake coppers are somewhat less corrosion resistant than the average. There does not appear to be any significant differences in corrosion resistance among the following types of copper: prime lake, silver-bearing lake, phosphorus-deoxidized electrolytic, fire-refined A.B.S., silver-bearing electrolytic, or arsenical electrolytic. There is some indication that the arsenical lake coppers are corroded more uniformly than the other coppers in industrial atmosphere.

In rural atmosphere, pure electrolytic, prime lake, silver-bearing lake and phosphorus deoxidized electrolytic coppers are corroded significantly more than the average; arsenical lake and arsenical electrolytic are corroded significantly less than the average, and there are no significant differences among fire-refined Wallaroo, fire refined A.B.S., and silver-bearing electrolytic copper. The effect of corrosion on electrical resistance confirms, in general, the weight-loss results at the rural location.

The arsenical coppers are significantly more resistant to clean sea water than the other coppers tested, as confirmed by both weight changes and changes in tensile strength.

The differences in resistance to atmospheric corrosion of the several types of copper are small in magnitude and probably have little, if any, practical significance in normal service.

Acknowledgment:

The authors are indebted to the personnel of the Analytical and Physical Testing Laboratories of The American Brass Co. for their assistance in obtaining some of the data.

DISCUSSION

Mr. F. L. LaQue.[1]—Some recent observations on electrolytic copper specimens partially immersed in sea water for a period of years have indicated that the rate of corrosion of copper decreases with time. This is in agreement with the data plotted in Fig. 10 of the paper. Our evidence of the decrease in corrosion rates was that during the first several months of exposure of the copper it corroded fast enough to keep itself clean so far as adherent fouling marine organisms were concerned, while more recently the copper has become completely fouled over the immersed portions, evidently due to the fact that the rate of corrosion had decreased below that required to prevent fouling.

There seems to be an apparently anomalous behavior of some of the coppers. It was noted on page 640 of the paper that the adherence of the corrosion products to arsenical coppers was much less firm than in the case of the other coppers. At the same time, the conclusion which was supported by the data was that these arsenical coppers were corroded more slowly than the other coppers. This would appear to be contrary to the general rule that the high-copper alloys which form loosely-adherent corrosion products are likely to be corroded by sea water to a greater extent than those which develop adherent corrosion-product films.

It was also noted on page 640 of the paper that there were big differences among the coppers with respect to edge attack. In observations of similar tension specimens of copper and high copper alloys immersed in flowing water we have frequently observed considerable erosion of upstream edges. It would be interesting to have the authors' comment on the possibility that the edge attack reported was due to some similar erosive effect of tidal action in addition to, or instead of, such more usual effects as those associated with the structure of the metal and the mechanical preparation of the edges of the specimens.

It is very interesting to find that statistical methods of analysis could be applied so usefully to the interpretation of data from tests which evidently were not designed originally to be subjected to statistical analysis. Apparently there is more latitude in the practical use of statistical methods than strict mathematical considerations would indicate.

In conclusion, the authors are commended for supplying so much useful data in a way that facilitates study and interpretation. The comparisons among the several types of copper should aid greatly in extending the usefulness of similar data being accumulated by Subcommittee VI on Atmospheric Corrosion of the Society's Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys.

Mr. A. W. Tracy,[2] (*author's closure*).—Mr. LaQue's observations that copper specimens exposed to sea water over a period of time became fouled with marine growths as the corrison rate decreased is interesting. Our specimens were still free from marine growths at the end of two years, but continuously-immersed specimens might conceivably have been fouled earlier than these alternately-immersed specimens.

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[1] Metallurgist, Development and Research Division, The International Nickel Co., Inc., New York, N. Y.

[2] Assistant Metallurgist, The American Brass Co. Waterbury, Conn.

In answer to Mr. LaQue's question on the effect of adherence of corrosion products *versus* corrosion rate, observations were made on the adherence of corrosion products on dry specimens. It is quite possible that the corrosion products were definitely adherent on all the types of copper when wet.

With regard to edge attack, it is possible that the motion of the water caused the edge-pitting. Specimen racks were fastened to a rock-filled crib which was part of a boat slip. There was little actual flow of water past the specimens. If the edge corrosion had been caused specifically by motion of the sea water we would expect rounded edges rather than pitted edges.

THE TOTAL-IMMERSION CORROSION TEST

By W. A. Wesley[1]

Synopsis

The total-immersion test is generally regarded as an unreliable type of corrosion test for which adequate means of control of the important variables are lacking. This belief is inconsistent with experience in routine and research testing of the corrosion of heavy metal alloys. Any test procedure is considered reliable which can be reduced to a state of statistical control. Multiple measurements of corrosion rates of monel and steel were made by a total-immersion procedure. The data furnish evidence of statistical control and the reproducibility of the results is considered adequate for both engineering and research purposes.

The principal details of procedure which draw comment are (1) the provision of an unusually high rate of aeration, (2) the value of exposing identical samples in separate jars rather than in the same jar of corrosive solution, (3) control of relative motion of solution and metal by mechanical movement of the specimen, and (4) removal of a layer of metal surface prior to test.

Total-immersion tests are indispensable in solving many corrosion problems which arise in the process industries and in developing alloys for equipment designed to handle corrosive liquids. Serious attempts have been made to standardize continuous immersion tests (**1**)[2] but most of these met with discouraging results. As a matter of logic, any attempt to develop a single procedure which would be reliable in testing a wide variety of metals in different types of corrosive media must be expected to fail. It is obviously impossible to control such variables as velocity, degree of aeration, metal surface condition, or even temperature over the full ranges encountered in practice by means of any one design of apparatus or test procedure. On the other hand, the mechanism of corrosion in many different metal-solution reaction systems is the same and it should be possible to standardize the control of influential variables within certain ranges to give procedures reliable for more than one metal-solution combination. A simple analogy is to be found in tension testing where one A.S.T.M. standard procedure serves for a wide variety of metals, and another is used in testing plastics.

An initial goal in standardization of total-immersion tests might well be the specification of any procedure which will yield reproducible results when it is applied to some one metal-solution combination by different qualified investigators. According to modern philosophy (**2**, **7**) there would be no point in having numerous investigators carry out comparison tests until the proposed procedure were refined to a state of statistical control. When this has been done and confirmed then the limits of applicability of the test procedure to other corrosion reaction systems should be determined and defined.

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[1] Assistant Director, Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 658.

Passano (3) demonstrated that measurement of the corrosion rate of iron exposed to a potable water can be properly controlled, but his problem was simplified by the availability of an unlimited source of water at constant temperature, degree of aeration, and pH value. Experience at Bayonne with a total-immersion test applied to nickel, iron, or copper alloys exposed to corrosive liquids of a wide variety of compositions has been good in that practical recommendations or developments based upon such tests over a period of 18 yr. have generally proved to be sound. The test procedure has been that described by Fraser, Ackerman, and Sands (4) who presented a thorough discussion and demonstration of the influence of controllable variables in total-immersion tests. It is not possible to apply an objective criterion to determine the reliability of the bulk of the data accumulated over these many years because the number of repeated observations made under essentially the same conditions was generally two and seldom over four. It so happens that one combination of alloy and solution was tested many times (and by different operators) so that it is possible to examine these results statistically.

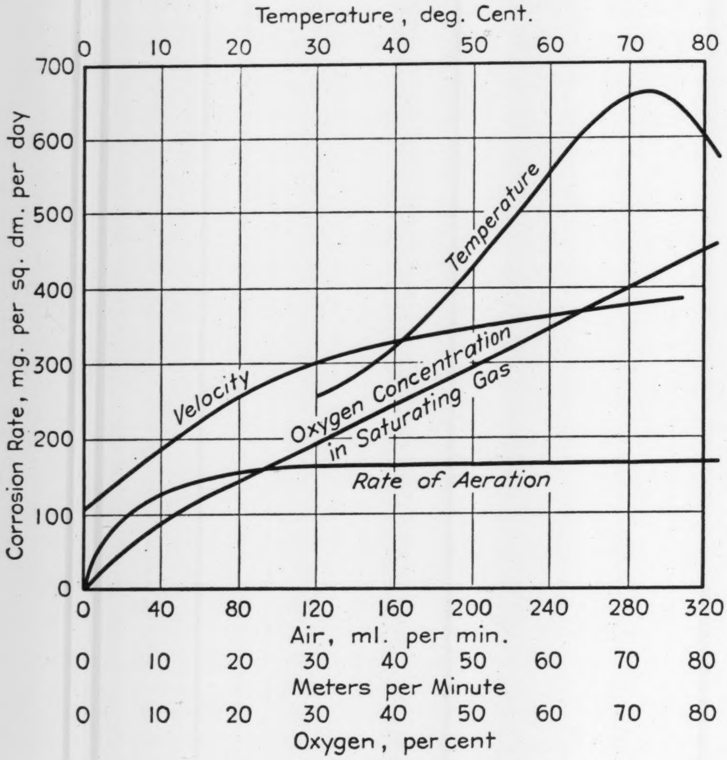

FIG. 1.—Influence of Controllable Factors.

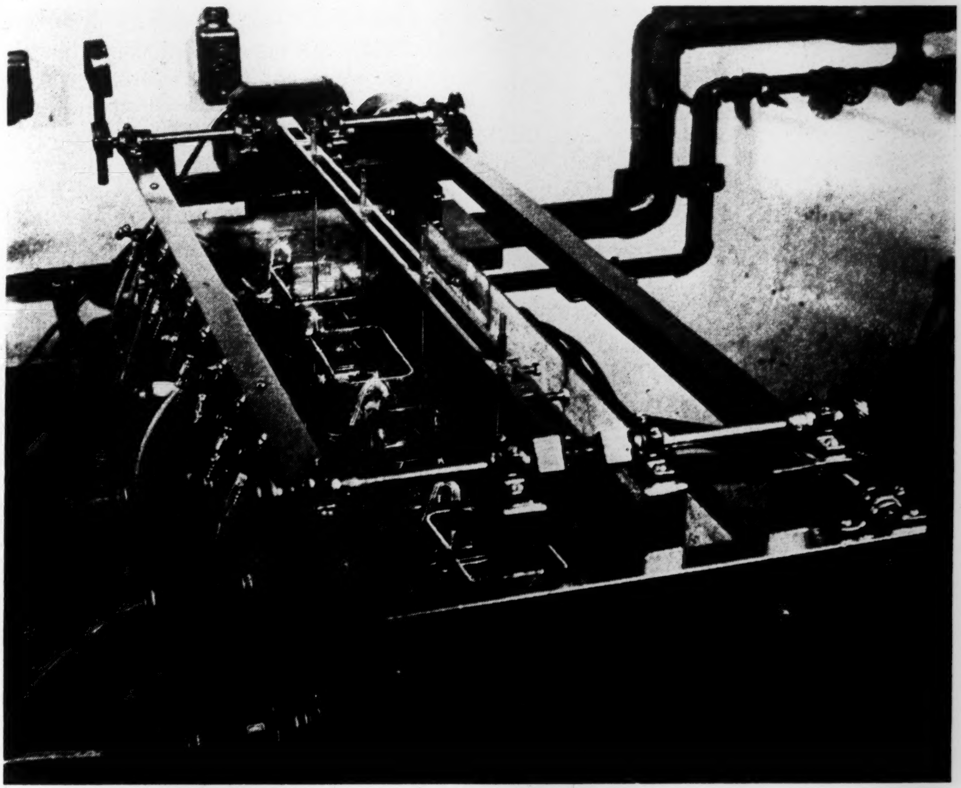

FIG 2.—Circular Path Corrosion Apparatus. Showing four jars in place.

THE RATE OF CORROSION OF MONEL IN AERATED 5 PER CENT SULFURIC ACID SOLUTION

Five per cent sulfuric acid solution (1.05 *N*) has been used as a medium representative of dilute, nonoxidizing mineral acid solutions whenever comparisons are to be made between the corrosion resistances of different grades of monel, different wrought forms, or different methods of fabrication. For this reason a large amount of laboratory information is available on the rate of corrosion of monel in this solution. The magnitude of the influence of some of the important controllable factors is shown in Fig. 1. It is obvious that at least the four variables shown must be controlled if a test of monel in aerated 5 per cent sulfuric acid solution is to have any permanent value.

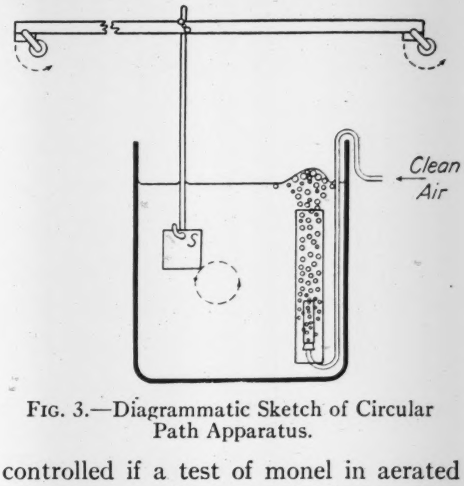

FIG. 3.—Diagrammatic Sketch of Circular Path Apparatus.

TABLE I.—NOMINAL CHEMICAL COMPOSITION OF WROUGHT MONEL.

Grade	Nickel, per cent[b]	Copper, per cent	Iron, per cent	Manganese, per cent	Silicon, per cent	Carbon, per cent	Sulfur, per cent	Aluminum, per cent
Regular	67	30	1.4	1.0	0.1	0.15	0.01	...
"R"[a] monel	67	30	1.7	1.1	0.05	0.10	0.035	...
"K"[a] monel	66	29	0.9	0.4	0.5	0.15	0.005	2.75

[a] Reg. U. S. Patent Office.
[b] Includes a small amount of cobalt.

The circular path apparatus (4) used in obtaining the data summarized in Table II is of the type illustrated in Figs. 2 and 3. Temperature was controlled thermostatically within the limits 30 ± 1 C., as measured within the sulfuric acid solution. The oxygen concentration in the saturating gas was constant since air was used. The velocity of movement of the test specimen in its vertical circular path through the solution was held constant during each series of tests but was not always set at the same value. The rate of aeration was not recorded in most tests but was judged by eye to be always above 100 ml. per min. per liter of solution, that is, in the range shown in Fig. 1 where variation in rate of aeration has little effect upon the corrosion rate. The air stream was introduced through an alundum thimble or crucible of medium porosity surmounted by a glass chimney which prevented impingement of air bubbles upon any specimen. Solution volume and the number of specimens per test jar were not standardized. Duplicate specimens were usually but not always tested in the same jar.

Variability with No Control of Metal:

The results of all tests which could be found in the laboratory records for monel of whatever grade or form, exposed to 5 per cent sulfuric acid solution at 30 C. and at velocities of close to 5 meters per minute, were assembled for study. Nominal analyses are given in Table I. Consider first the over-all picture presented in the last line of Table II: no matter what grade of monel, what surface finish, what year produced, what operator or what duration of test, the observed rate of corrosion was within the limits 129 to 435 mg. per sq. dm. per day. Perusal of the literature on total immersion tests would certainly lead one to expect greater spread due to variability in the quality alone of samples of a commercial metal. Indeed, these limits could be considered almost narrow enough to permit use of the average rate for design of process equipment, since uncertainty in the ranges of variables such as oxygen content of the solution, relative velocity

TABLE II.—DATA ON CORROSION OF MONEL IN 5 PER CENT SULFURIC ACID.

Temperature.................... 30 ± 1 C.
Aeration........................ More than 100 ml. air per min. per liter of solution
Velocity......................... 4.7 to 6.4 meters per min.
Duration........................ 20 hr. to 32 days.

Grouping	Years	Number of Operators Represented	Number of Observations, *n*	Weight Loss, mg. per sq. dm. per day				
				Average, $\bar{X}$	Standard Deviation, σ	Max.	Min.	Range, *R*
Rod specimens	1926–1941	5	36	260.3	29.0	336	215	121
Tube specimens	1938–1941	2	22	275.6	30.9	326	189	137
Plate specimens	1938–1941	1	16	280.9	37.7	350	236	104
Sheet specimens	1927–1942	3	73	266.2	57.0	435	129	306
Sheet (control test only)	1942	1	24	235.5	17.9	263	189	74
"K" monel only	1935 and 1941	2	14	242.3	25.0	271	186	85
"R" monel only	1936	1	8	224.0	9.3	241	209	32
All tests	1926–1942	9	161	263.9	46.7	435	129	306

of metal *versus* solution and actual time of contact with the solution during its over-all period of use, makes it impractical to be more precise in estimating the probable expense of metal loss by corrosion.

Further encouragement was obtained upon grouping the data in various ways. This showed that the wide over-all range was not due to any significant trend in average results during the years nor with different durations of test but was due primarily to variability in some characteristic of sheet specimens. As shown in Table II the ranges for other wrought forms of monel were less than one half the over-all range.

Reproducibility with Control of Quality of Metal:

Examination in detail of the earlier data suggested a change in procedure with regard to the number of specimens exposed within a given test jar. The practice of testing more than one specimen per jar gives a misleading improvement in agreement between such specimens, due to the fact that the specimens in a given jar are subjected to exactly the same cycles of chance variation in degree of aeration, temperature, and velocity. In other words, putting four "identical" specimens in one jar may not be equivalent to repeating an experiment four times and should not give the same statistics as would be obtained by four repetitions of the measurement on successive days. Thus, in spite of the wide range of the data from which Table II was compiled, there were 13 pairs of specimens, each pair exposed in one jar, for which the difference in the duplicate corrosion rates was not more than 1 per cent![3]

To establish the reproducibility of the modified total-immersion test procedure, experiments were performed in which specimens were cut from a single sheet of cold-rolled monel and were exposed in groups of four on successive days. One test specimen 5 cm. square by 0.079 cm. thick with a single hole for suspension on a glass hook was exposed in each glass jar containing 2 liters of 5.0 per cent sulfuric acid solution. Four experiments were performed simultaneously, and since it was unfortunately not convenient to control the temperature by placing the jars in separate thermostats, all four jars were placed in one thermostat. Preliminary tests demonstrated that the corrosion rate of the sheet specimens decreased in successive tests until a surface layer about 0.0003 cm. thick had been removed by corrosion or resurfacing. The pieces used in the present tests were, therefore, pickled in aerated dilute sulfuric acid solution until at least this amount of metal had been

TABLE III.—CORROSION OF MONEL IN 5 PER CENT SULFURIC ACID WITH CONTROL OF METAL QUALITY.

Composition of monel	nickel 67.16, copper 30.85, iron 0.69, manganese 1.05, silicon 0.06, sulfur 0.005 per cent.
Temperature	30 ± 1.5 C.
Velocity	4.8 meters per min.
Aeration	About 200 ml. air per liter per min.
Duration	20 hr.
Area of monel	0.507 sq. dm. per specimen.
Volume of solution	2 liters.

Corrosion Rate, mg. per sq. dm. per day				Average, $\bar{X}$	Standard Deviation, σ
X_1	X_2	X_3	X_4		
251.8	252.1	263.2	232.7	249.95	10.97
260.6	239.5	255.4	217.0	243.13	16.96
229.8	238.8	231.5	247.8	236.98	7.08
232.7	220.6	225.3	188.6	216.80	16.84
236.7	254.7	251.4	244.3	246.78	6.89
226.0	232.4	214.9	203.3	219.15	11.09
Average				235.47	11.64

CENTRAL LINES

For $\bar{X}$: $\bar{\bar{X}} = 235.47$
For σ: $\bar{\sigma} = 11.64$

CONTROL LIMITS

For $\bar{X}$: $235.47 \pm (1.88)\ 11.64 = 257.35$ and 213.59
For σ: 0 and $(2.33)\ 11.64 = 0$ and 27.1

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[3] The same reasoning leads to the conclusion that it may be advantageous to immerse two different specimens (not duplicates) in the same jar in comparing corrosion rates, provided that the corrosion products of one material have no effect upon the rate of corrosion of the other.

removed. They were then polished with No. 240 carborundum paper, degreased by scrubbing with clean pumice powder, rinsed in clean water, and dried with a clean towel. Aeration was controlled by introducing filtered air through a sintered Pyrex glass diffusing disk of medium porosity and 2 cm. radius. The rate of flow of air was not measured in each experiment but was set by eye at an amount estimated to be about 400 ml. per min. or 200 ml. air per liter of solution per minute.

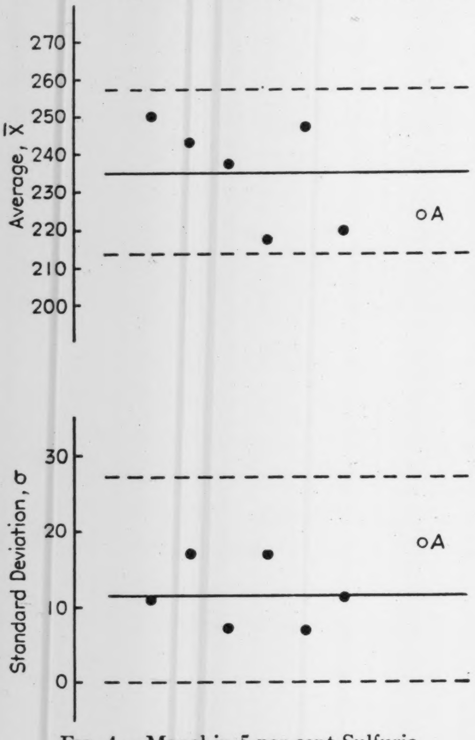

FIG. 4.—Monel in 5 per cent Sulfuric Acid Solution.

The results of six groups of four observations each are presented in Table III. Control charts (5) are reproduced in Fig. 4. Although a positive demonstration of a state of statistical control is said (2, p. 37) to require that the averages from at least 25 successive groups of four observations shall lie within the control limits, the above data do offer substantial evidence of control.

Passano (3) pointed out that it is not necessary to standardize apparatus in standardizing a total-immersion test. The same results should be obtained in any apparatus in which the required conditions are established and maintained. To support this statement an additional group of four successive corrosion tests was performed by R. F. Stearn[4] using a vertical spindle apparatus

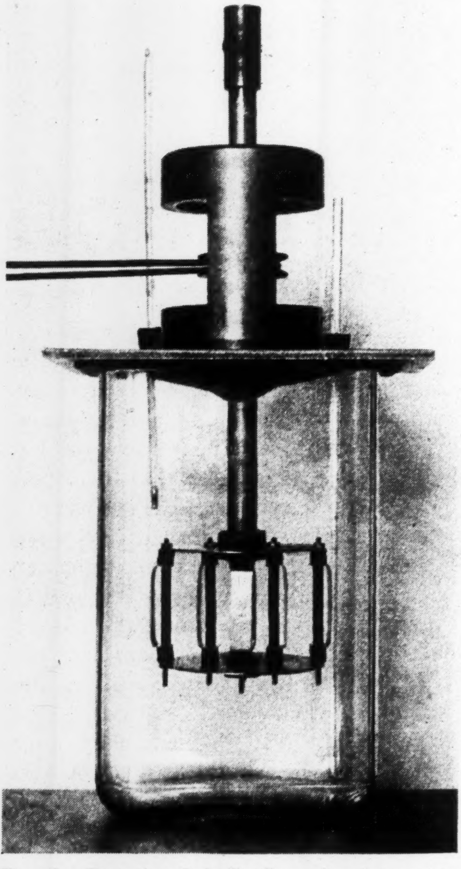

FIG. 5.—Rotating-Spindle Corrosion Apparatus.

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[4] Unpublished Data, Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

to maintain a constant velocity of 4.9 meters per minute. The apparatus is illustrated in Fig. 5 but only one specimen was tested at a time. The solution volume in these tests was 4 liters and aeration was at a measured rate of 200 ml. air per min. His results are plotted in Fig. 4 as points marked *A*. They fall well within the control limits established for the data obtained in the circular-path apparatus, both in respect to the average and the standard deviation.

TABLE IV.—CORROSION OF MILD STEEL IN 3 PER CENT SODIUM CHLORIDE SOLUTION.

Composition of steel	copper 0.09, nickel 0.08, sulfur 0.038, manganese 0.32, phosphorus 0.012, carbon 0.07, silicon less than 0.01 per cent.
Temperature	30 ± 1.5 C.
Velocity	4.8 meters per minute.
Aeration	About 400 ml. air per liter per min.
Duration	20 hr.
Area of Steel	0.539 sq. dm. per specimen.
Volume of solution	2 liters.

Corrosion Rate, mg. per sq. dm. per day				Average, $\bar{X}$	Standard Deviation, σ
X_1	X_2	X_3	X_4		
246.9	227.1	245.8	248.7	242.13	8.74
231.8	235.8	211.1	217.7	224.10	10.08
220.4	216.4	221.7	233.1	222.90	6.21
218.2	240.9	236.9	244.7	235.18	10.16
257.4	252.5	248.1	240.6	249.65	6.18
252.7	247.3	238.0	239.1	244.28	6.01
Average				236.37	7.90

CENTRAL LINES

For $\bar{X}$: 236.37
For σ: 7.90

CONTROL LIMITS

For $\bar{X}$: $236.37 \pm (1.88)\ 7.90 = 251.22$ and 221.52
For σ: 0 and $(2.33)\ 7.90 = 0$ and 18.41

RATE OF CORROSION OF STEEL IN AERATED 3 PER CENT SODIUM CHLORIDE SOLUTION

It was desired to determine the reproducibility of the total-immersion test in measuring rates of corrosion with a decidedly different combination of metal and corrosive solution. Eighty test specimens were cut from a sheet of mild steel. These were shuffled and the 24 specimens used in corrosion tests were drawn at random from the lot. Specimen preparation consisted of pickling in aerated 10 per cent hydrochloric acid solution at room temperature for about 80 min., followed by polishing with No. 240 carborundum paper, scrubbing with clean pumice powder and drying upon a clean towel. The pieces were weighed immediately and entered into the test jars within 20 min. of the time at which they had been cleaned. There was only one specimen in each jar containing 2 liters of 3 per cent sodium chloride solution (0.52 *N*). Four jars were immersed in a thermostat at one time and six successive groups of four specimens were tested. Analyses of samples of the sodium chloride solution removed just before the end of several tests showed an average oxygen concentration of 0.00073 normal as compared with 0.00078 normal for the same solution when fully saturated with air at 30 C.

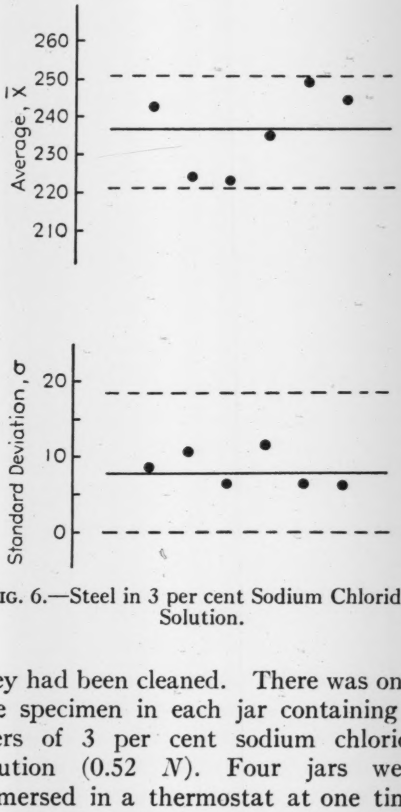

FIG. 6.—Steel in 3 per cent Sodium Chloride Solution.

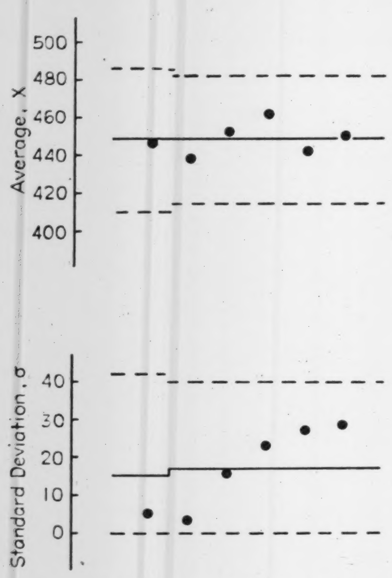

FIG. 7.—Monel in 5 per cent Hydrochloric Acid.

At the end of the corrosion test the specimens were cleaned by rubbing them with the fingers with a small amount of soap and powdered pumice. This type of cleaning was effective in removing corrosion products yet required no correction for loss due to metal removal. It should be noted that the specimens were not attacked over all the surface but that anodic areas were irregularly distributed with from 25 to 40 per cent of the surface apparently unattacked.

The results are presented in Table IV and Fig. 6. As in the previous measurements with monel the statistics on steel fall within the control limits.

MONEL IN 5 PER CENT HYDROCHLORIC ACID SOLUTION

Control charts for measurements of the rate of corrosion of monel in aerated 5 per cent hydrochloric acid solution (1.4 *N*) are given in Fig. 7 from data obtained by B. B. Knapp and R. F. Stearn.[4] There were three results in the first group and four in each of the others. A circular-path apparatus was used at a velocity of 4.5 meters per minute. The duration of test was 24 hr. and the temperature was 30 ± 1 C. Aeration was at the rate of 375 ml. per liter of solution per minute. One specimen of area 0.51 sq. dm. was exposed in each jar of 4 liters of solution.

COPPER IN 1 *N* SULFURIC ACID SOLUTION

B. B. Knapp and R. F. Stearn[4] have attempted to apply the above procedure to the corrosion of copper in aerated sulfuric acid solution but have not succeeded in obtaining statistical control. The problem has not yet been solved but suspicion is attached to two causes of variability. First, the corrosion rate is much higher than those measured in the present work so that even a maximum rate of air flow through one diffuser may be inadequate to maintain the solution saturated with air. Second, there are indications that copper ions in solution tend to accelerate the

TABLE V.—SUMMARY OF STATISTICS OF CONTROLLED CORROSION TESTS.

	Number of Observations	Average Loss, $\bar{X}$, mg. per sq. dm. per day	Average Standard Deviation (n = 4), $\bar{\sigma}$	Universe Standard Deviation, σ'	Universe Coefficient Of Variation, v', per cent	Error of the Average P = 0.95[a]	
						mg. per sq. dm. per day	per cent
1. Monel in 5 per cent sulfuric acid.......	24	235.47	11.6	14.6	6.2	±5.84	±2.5
2. Steel in 3 per cent sodium chloride.....	24	236.37	7.90	9.90	4.2	±3.96	±1.7
3. Monel in 5 per cent hydrochloric acid..	23	448.60	17.2	21.6	4.8	±8.85	±2.0

[a] Error of average = $\frac{Z\sigma'}{\sqrt{N}}$; where Z = 1.96 when P = 0.95.

corrosion rate. It may be necessary to introduce a definite concentration of copper into the acid at the start in order that this factor may remain substantially constant during the corrosion test.

NUMBER OF REPETITIONS

The statistics of the three sets of controlled corrosion tests summarized in Table V permit an interesting calculation, namely, what number of multiple experiments should be made in applying this total-immersion testing procedure? Of course, no attempt should be made to apply the conclusions that are to be drawn from these statistics to corrosion reactions which differ much in mechanism from those of Table V since it is necessary to assume a coefficient of variation comparable to those in the table.

Assuming that the coefficient of variation will not be greater than 6.2 per cent, the expected limits of error of the average of groups of various sizes can be estimated for a statistical probability of 0.95 as follows (6):

$$\text{Error } E = \frac{Z\sigma'}{\sqrt{N}}$$

Expressed in percentage of the average

$$E = \frac{Zv'}{\sqrt{N}} \text{ per cent}$$

The value of Z for $P = 0.95$ is 1.96. Therefore, the expected errors of the averages for different group sizes would be:

$N = 8 \quad E = \pm\ 4.3$ per cent
$N = 4 \quad E = \pm\ 6.1$ per cent
$N = 2 \quad E = \pm\ 8.6$ per cent

From these results it can be seen that running the total-immersion tests in quadruplicate makes it highly probable that the average will be within 6 per cent of the true average. This can be considered quite satisfactory precision for most corrosion research programs. On the other hand, performing each test four times in order to attain this precision would generally be considered not worth the cost in routine determinations which are made to aid in choice of materials of construction or in making an engineering estimate of the necessary wall thickness of a piece of apparatus. For much of this sort of work limits of ±10 per cent are small enough in view of the variability of other factors affecting the recommendations drawn. Although estimation of the error of average of but two measurements is not statistically sound, there does seem to be some justification for the prevailing practice of running routine total-immersion tests in duplicate only.

COMMENTS ON TEST PROCEDURE

The principal differences between the procedure employed in the tests described above and that ordinarily followed in attempts to standardize a total-immersion test are the following:

(*a*) A high rate of aeration was used in the present work, namely, 200 to 400 ml. air per minute per liter of solution, introduced through a porous thimble or disk. This is in contrast with very low rates of aeration or even failure to use a diffusing device in other procedures. In order to secure adequate control it is probably necessary to maintain the concentration of the aerating gas in the corrosive solution near the saturation point. If it is desired to maintain the oxygen concentration at a value different from the point of saturation with air, this can be better accomplished by altering the composition of the saturating gas rather than by altering the rate of flow of the gas.

(*b*) Duplicate specimens should be tested in separate jars. Testing multiple specimens in a single jar yields values of standard deviation for such subgroups which may not be equivalent to the true variability of the corrosion test as determined by repeated separate tests.

(*c*) Control of the relative motion of solution and specimen was obtained in the present tests by mechanical movement of the specimen, as contrasted with motion produced solely by the air stream introduced into the solution.

(*d*) Variation due to the quality of the surface of sheet metal test specimens may be reduced by removal of a substantial layer of the original metal surface prior to the corrosion test. In the present experiments a layer at least 0.0003 cm. thick was removed by pickling.

It should be remarked that no attempt has been made in this report to interpret the influence of the many other factors which can affect the magnitude of corrosion rates, such as duration of test, volume of solution used and influence of the corrosion products. These were presumably held constant and attention has been confined to the reproducibility of the method itself as applied to three corrosion reactions.

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(2) W. A. Shewhart, "Statistical Method from the Viewpoint of Quality Control," Washington, The Graduate School, Department of Agriculture (1939).

(3) R. F. Passano, "Controlled Data from an Immersion Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 468 (1932).

(4) O. B. J. Fraser, D. E. Ackerman and J. W. Sands, "Controllable Variables in the Quantitative Study of the Submerged Corrosion of Metals," *Industrial and Engineering Chemistry*, Vol. 19, p. 332 (1927).

(5) " 'Control Chart' Method of Analysis and Presentation of Data," Supplement B (1935) to A.S.T.M. Manual on Presentation of Data, *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1418 (1935).

(6) C. H. Humes, R. F. Passano and Anson Hayes, "A Study of the Error of Averages and Its Application to Corrosion Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 448 (1930).

(7) W. E. Campbell, "The Use of Statistical Control in Corrosion and Contact Resistance Studies," *Transactions*, Electrochemical Soc., Vol. 81, p. 377 (1942).

DISCUSSION

Mr. A. W. Tracy[1] (*presented in written form*).—In this paper, as in previous papers by Mr. Wesley, he has presented data on a fundamental problem pertaining to corrosion clearly and concisely. The members of Subcommittee I on Total Immersion Test of the Society's Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, who drew up the Proposed Tentative Method of Total Immersion Testing of Non-Ferrous Metals presented at this meeting,[1a] are indebted to Mr. Wesley for numerous ideas which were incorporated in that tentative method.

I should like to ask Mr. Wesley whether he has any data to indicate whether corrosion results will be reproducible when the specimen has nominally zero velocity relative to the corroding solution, or if currents set up by convection and aeration cause lack of reproducibility.

Mr. R. H. Brown[2] (*presented in written form*).—The careful investigation that has been carried out by Mr. Wesley in order to bring the total-immersion test under statistical control is evident from the data that have been submitted in this paper. The methods of analysis are demonstrated and will probably serve as a model for others to follow.

It is stated in the paper that it is not necessary to standardize apparatus in standardizing the total-immersion test, provided the required conditions are established and maintained. The qualifying clause which requires the establishment and maintenance of the required conditions would, in one sense, require standardization of the equipment. Furthermore, it is often extremely difficult to evaluate those factors which control the corrosion rate under service conditions. Consequently, the total-immersion tests of the type described by Mr. Wesley would appear to be useful largely in establishing quality control of material in order to meet certain specifications. For example, Mr. Wesley has pointed out that it may be necessary to add copper ions to the solution used in the total-immersion test in order to bring tests on copper under statistical control. However, under service conditions the corroding environment may not contain these copper ions, and consequently the corrosion measured in the total-immersion corrosion test would not necessarily apply to the design of commercial equipment using copper.

It might be pointed out with regard to the statistical analysis of the data, that it should be possible to reduce the standard deviation, even to a greater extent than has been so far accomplished, by even closer control of the various factors mentioned by Mr. Wesley, and possibly others.

It may be possible, by the statistical methods described by Mr. Wesley, to study certain factors in the mechanism of corrosion. The example he has cited in the case of corrosion of copper, whereby the test may be brought under statistical control by addition of copper ions to the solution, demonstrates that their presence alters the corrosion rate. Automatically, then, those factors which are varied in order to bring the test under statistical control also must be involved in the mechanism of corrosion (provided the method of measurement is under statistical control).

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[1] Assistant Metallurgist, The American Brass Co., Waterbury, Conn.

[1a] This method was accepted as tentative by the Society and is published as Tentative Method of Total Immersion Testing of Non-Ferrous Metals (B 185 - 43 T), 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 332.

[2] Research Electrochemist, Aluminum Company of America, New Kensington, Pa.

MR. W. A. WESLEY[3] (*author's closure, by letter*).—I thank Messrs. Brown and Tracy for their discussion. Mr. Brown's pessimism regarding the possibility of estimating service total immersion corrosion rates from data obtained in a controlled total immersion laboratory test is in contrast to our optimism. Both are undoubtedly based upon experience, in the former case with aluminum alloys, in the latter with heavy metals. There is a marked general difference in the types of corrosion and in the relative importance of corrosion-controlling factors involved in the commercial uses of the two groups of materials. In this probably lies the cause of disagreement.

To Mr. Tracy's question I must reply that the only extensive attempt we have made to evaluate the reliability of data obtained when the corrosion specimen is not moved was one connected with Knapp and Stearn's study of copper corrosion which was quoted in the paper. They found a high rate of corrosion of copper in sulfuric acid solution accompanied by a lack of statistical control. Now, when tests were run with the specimen held stationary, a much lower rate of corrosion was observed and there was a relatively high standard deviation but the control chart method of analysis showed statistical control! The results can be summarized as follows:

CORROSION OF COPPER IN AERATED 1 *N* SULFURIC ACID SOLUTION.

Temperature: 35 C.
Aeration: 1000 ml. per min.

	Motion Meters per min.	Number of Observations, N	Average Loss, mg. per sq. dm. per day, $\bar{X}$	Standard Deviation, σ	Statistical Control
With motion.	4.8	20	558.1	44.7	No
Without motion....	0	20	276.8	55.9	Yes

Against these data can be cited the experience of Subcommittee I on Total Immersion Testing of the Society's Committee B-3 which has sponsored multiple tests performed in different laboratories by a procedure involving stationary test specimens. The results proved lack of statistical control.

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[3] Assistant Director, Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

THE TECHNICAL COHESIVE STRENGTH AND OTHER MECHANICAL PROPERTIES OF METALS AT LOW TEMPERATURES

By D. J. McAdam, Jr.,[1] and R. W. Mebs[1]

Synopsis

The paper first presents and discusses diagrams representing the technical cohesive strength of metals at room temperature and at selected low temperatures. Comparison of these diagrams shows the influence of low temperatures on the form of the diagram and gives a basis for interpretation of the influence of low temperature on the strength, ductility, and total work of notched and unnotched specimens.

A study has also been made of the quantitative variation of the technical cohesive strength and other mechanical properties. This variation is studied with reference to the usual arithmetical scale of temperatures, and also with reference to a temperature scale of equal thermodynamic efficiencies. The use of the latter scale reveals relationships of considerable theoretical and practical importance.

The technical cohesive strength of metals has been discussed in three previous papers by one of the authors (**11, 12, 13**)[2] and in a recent paper by both authors (**14**). As shown in these papers, the technical cohesive strength of a metal, in any particular state as regards prior mechanical treatment and heat treatment, cannot be represented by a single value of the algebraically greatest principal stress.[3] The *technical cohesive strength* of any metal comprises an infinite number of *technical cohesion limits*, each corresponding to a specific combination of the principal stresses. The technical cohesive strength of a metal, therefore, must be represented by a diagram consisting of curves of variation of the cohesion limit with variation of the stress system. Such diagrams of various types have been presented in the previous papers (**11, 12, 13, 14**). Diagrams were also presented to represent the influence of plastic deformation on the technical cohesive strength. These diagrams show that plastic deformation causes continuous increase in the technical cohesive strength.

In the previous papers, attention was confined to the technical cohesive strength of metals at room temperature. In this paper, the view is broadened to include an additional variable, temperature. Attention is first directed to the

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[1] Metallurgist, and Associate Metallurgist, respectively, National Bureau of Standards, Washington, D. C.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 702.

[3] Any stress system may be resolved into three principal stresses normal to three mutually perpendicular planes, known as the "principal planes," on which there is no shear. Tensile stresses are viewed as positive, and compressive stresses are viewed as negative. In this paper as in the previous papers, the algebraically greatest principal stress will be designated S_1, the least principal stress will be designated S_3, and the intermediate principal stress will be designated S_2.

influence of low temperature on the form of the diagrams representing technical cohesive strength. Diagrams representing the technical cohesive strength of some metals at selected low temperatures are compared with corresponding diagrams representing the technical cohesive strength at room temperature. A study is then made of the quantitative variation of the technical cohesive strength, ultimate stress, yield stress, and ductility throughout a range of temperature from that of liquid air (−188 C.) to +165 C. Diagrams of several types are used to show this variation. All these diagrams are based on results of tension tests of notched and unnotched specimens.

Very few thorough investigations have been made of the influence of a wide range of low temperatures on the strength of metals. A number of investigators have studied the yield strength and ultimate tensile strength of metals at selected low temperatures, such as the temperatures of solid carbon dioxide (−78 C.) or liquid air (−188 C.), but have not presented curves of variation of these properties with temperature. A few investigators have presented curves of variation of ultimate stress throughout a range of temperature from −188 C. to room temperature or higher. No previous investigations appear to have been made of the influence of low temperature on the "true" breaking stress of unnotched specimens, or on the ultimate stress and breaking stress of notched specimens.

METHOD OF INVESTIGATION, SPECIMENS, AND MATERIALS

The method of investigation of the technical cohesive strength is based on the fact that when a notched cylindrical specimen is subjected to longitudinal tension, the minimum cross-section is under transverse radial tension. The mean radial stress tends to increase with the depth and sharpness of the notch, but is always less than the longitudinal stress. The algebraically greatest principal stress (S_1), therefore, is longitudinal. The radial stress is equivalent to two equal principal stresses (S_2 and S_3) which are rotatable without change around the axis of the specimen. By the use of notched specimens, therefore, it is possible to investigate the influence of the radial stress ratio (S_3/S_1) on the technical cohesion limit, ultimate stress, yield stress, and ductility. One of the previous papers (**14**) gives results so obtained with both shallow and deep notches. In the present paper, attention is confined to the influence of deep notches. With such notches the disturbing effects of stress concentration are minimized, and thus do not interfere with a study of the influence of the radial stress ratio (**14**).

Most of the notched specimens were approximately 6 in. in length; those having large angles were necessarily somewhat longer. The ends of all specimens were threaded to $\frac{3}{4}$ in. in diameter. Between the threaded ends, each specimen was machined to a selected uniform diameter and a circumferential V notch of fixed angle, depth, and root radius was machined at the mid-section. The notch radius was carefully machined so that the conical sides of the notch were tangent to the arc at the root. Care was also taken to avoid heating, bending, or appreciable cold-working during machining.

Before testing, each specimen was carefully calibrated as follows: The maximum diameter was measured by micrometer calipers; the diameter of the minimum section, at positions 90 deg. apart circumferentially, was determined by means of a measuring microscope.

The root radius was measured by the use of a graduated set of wires of slightly different diameters, and the notch angle was measured on a magnified photograph of the notched specimen.

The metals used to obtain the cohesion diagrams were oxygen-free copper, monel metal, 13 per cent chromium, 2 per cent nickel steel, low-carbon steel, and ingot iron (open-hearth iron). Additional metals were used in the investigation described in the Section on The Variation of the Strength of Metals Between Room Temperature and −188 C.

TABLE I.—DESCRIPTION OF METALS.

Metal	Designation	Mechanical Treatment		Bar Diameter, in.	Carbon, per cent	Iron, per cent	Nickel, per cent	Copper, per cent	Chromium, per cent	Aluminum, per cent	Tin, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent
		Method	Reduction of Area, per cent												
Oxygen-free copper	N	cold-rolled	75	0.875	...	...	...	99.97	...	...	...	...	...	...	...
Monel metal	G	cold-drawn	40	0.875	0.18	1.24	Diff.	28.46	...	...	...	0.94	...	0.007	0.10
Nickel	R	cold-drawn	60	0.625	0.07	0.08	99.49	0.03	...	...	...	0.24	...	0.005	0.08
Chemically pure aluminum	AA	cold-drawn	...	0.875	...	...	...	...	...	99.97	...	...	...	...	...
Commercial aluminum	AB	cold-drawn	...	0.875	...	...	...	...	...	99.4	...	...	...	...	...
Copper-tin alloy	BA	cold-drawn	...	0.750	...	<0.01	...	Diff.	...	...	3.9	...	0.3	...	...
Ingot iron	A	cold-drawn	...	0.750	0.015	Diff.	...	...	...	...	...	0.05	0.003	0.025	<0.01
0.04 per cent carbon steel	Q	cold-drawn	48	0.713	0.045	Diff.	...	...	...	...	...	0.04	0.003	0.03	<0.01
0.20 per cent carbon steel	T	hot-rolled	...	1.125	0.20	Diff.	...	...	...	...	...	0.42	0.039	0.049	0.21
3.5 per cent nickel steel	Z	hot-rolled	...	0.625	0.34	Diff.	3.30	...	...	...	...	0.72	0.018	0.017	0.26
13 per cent chromium, 2 per cent nickel steel	E	hot-rolled	...	0.875	0.09	Diff.	2.06	...	13.3	...	...	0.48	...	...	0.26

The oxygen-free, high-purity copper was supplied by the Scomet Engineering Co. through the cooperation of Sidney Rolle, Assistant Manager. It had been given a reduction of 75 per cent by cold rolling without intermediate annealing. Some of this material was tested in that condition and some was annealed at 800 F. The aluminum was supplied through the cooperation of the Aluminum Company of America.

The monel metal and nickel were supplied by the International Nickel Co. through the cooperation of A. J. Wadhams, Director of Research. The monel metal had been reduced to 40 per cent, by cold drawing without intermediate annealing. Some of this material was tested in that condition and some was annealed at 1400 F. in order to place it in its softest condition.

The 13 per cent chromium, 2 per cent nickel steel was obtained from the Carpenter Steel Co. through the cooperation of G. V. Luerssen. It was in its softest condition, which is obtained by heating to 1240 F. and cooling slowly in the furnace. This material was tested as received. The low-carbon steel and ingot iron, purchased in the open market, had been cold drawn to a reduction of 12 per cent without intermediate annealing. In this laboratory the low-carbon steel was cold drawn further to a total reduction of 48 per cent. Some of it was then heated to 1750 F. and cooled in the furnace.

All the metals were in the form of round rods. A description of the metals is given in Table I, and details of the heat treatments and mechanical treatments applied in the laboratory are given in Table II. For a description of the apparatus and method of test, reference should be made to a preceding paper (**14**).

To obtain the desired temperature, the specimen was fully submerged in an appropriate liquid contained in a brass vessel. The lower threaded end of the specimen passed through the bottom of the deep bowl-shaped vessel into the lower adapter of the testing machine. Felt washers were used to seal against loss of the bath liquid. The upper and lower adapters were made as long as the clearance of the testing machine would permit. A cylindrical shell of sheet aluminum surrounded the vessel, and conical shields of this metal extended from this cylinder to the head and base of the testing machine. When the test temperature was to be below that of solid CO_2, the temperature gradient in the adapters was minimized by surrounding the head and base of the testing machine with solid CO_2.[4]

TABLE II.—HEAT TREATMENT IN LABORATORY.

Material	Designation	Temperature, deg. Fahr.	Time Held, hr.	Cooled in	Temperature, deg. Fahr.	Time Held, hr.	Cooled in
Copper	N-8	800	5	...	...	...	...
Monel	G-14	1400	1	Water	...	...	...
Ingot iron	A-17.5	1750	1	Furnace	...	...	...
0.04 per cent carbon steel.	Q-17.5	1750	1	Furnace	...	...	...
0.20 per cent carbon steel.	T-W-9	1625	1	Water	900	2	Air
3.5 per cent nickel steel..	Z-O-10	1475	½	Oil	1000	1	Air

To obtain a temperature of −78 C., a mixture of carbon tetrachloride and chloroform was cooled by pulverized solid CO_2. To obtain temperatures between −78 C. and the temperature of liquid air (−188 C.), the bath liquid was the compound CCl_2F_2, which is in commercial use as a refrigerant. The boiling point is −29 C. and the melting point is −160 C. This liquid was cooled by pouring liquid air into an end of an immersed copper coil; the liquid being constantly stirred. A chromel-alumel thermocouple was attached to the specimen. The desired temperature could thus be accurately maintained.

To avoid local elevation of the temperature during plastic deformation, the tensile load was applied more slowly than is usual in a tension test. In tests of annealed single-phase metals, which extend considerably before local contraction, the time required for this extension was generally 15 min. or more. Slow loading is important because work hardening increases with decrease in temperature. Satisfactory results, consequently, cannot be obtained by loading rapidly at first, and then decreasing the rate before the load reaches the maximum.

The yield stress for unnotched specimens was approximately equivalent to a 0.1 per cent proof stress (stress producing 0.1 per cent plastic extension) as measured with a sensitive optical extensometer. For notched specimens at room temperature, the yield load was selected as the load giving an initial extension as indicated by the stress-strain record. For unnotched specimens at low temperatures, no stress-strain record was obtained, but a load-time curve was obtained. For ingot iron, 0.04 per cent carbon steel, annealed monel metal, and some other metals, the yield stress could thus be obtained with fair accuracy.

The strain corresponding to the initially recorded extension was somewhat greater locally for a notched specimen than for an unnotched specimen, and probably was about 3 per cent (**14**).

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[4] Valuable advice regarding this procedure was obtained from J. W. Cook of the Cryogenic Laboratory, National Bureau of Standards.

This difference should be kept in mind in comparing yield stress values for notched and unnotched specimens.

THE INFLUENCE OF LOW TEMPERATURE ON THE FORMS OF THE DIAGRAMS REPRESENTING TECHNICAL COHESIVE STRENGTH, ULTIMATE STRENGTH, AND YIELD STRENGTH

As shown in the previous papers (**11, 12, 13, 14**) neither the technical cohesive strength, ultimate strength, nor yield strength of a metal can be represented completely by a single stress value, but must be represented by a diagram showing the variation of stress values with the combination of principal stresses. When no two of the principal stresses are equal, three-dimensional diagrams must be used (**12**). In this paper, however, attention will be confined to stress combinations with two of the principal stresses kept equal. Such stress systems would be produced by subjecting a cylinder to various combinations of axial and uniform radial stress. The uniform radial stress may be resolved into two equal principal stresses, whose mutually perpendicular directions are rotatable around the specimen axis. (In such a system, the stress is the same in any direction perpendicular to the axis.) The influence of such stress systems on the strength of metals, therefore, can be represented by two-dimensional diagrams, with coordinates representing the axial and radial stresses.

Combinations of uniform radial tension with axial tension cannot readily be obtained experimentally. When a notched cylindrical specimen is subjected to longitudinal tension, however, the minimum section is under radial tension. The ratio of the mean radial stress to the axial stress increases with the depth and sharpness of the notch, but cannot become equal to 1.0. The radial stress, therefore, is S_3 and the axial stress S_1. The use of notched specimens in an investigation of the influence of the radial stress ratio (S_3/S_1) on the technical cohesion limit, ultimate stress, and yield stress has certain disadvantages due to the influence of the stress gradient induced by a notch. A stress gradient tends to make the estimated mean stress for any strength index somewhat too low. Slight plastic extension, however, greatly reduces the stress gradient. By the use of suitably designed notched specimens, therefore, it is possible to investigate the influence of the radial stress ratio on the technical cohesion limit and ultimate stress, and even on the yield stress (**14**).

The Influence of the Notch Angle and Root Radius on the Yield Stress and Ultimate Stress:

In a study of the technical cohesive strength of metals by means of notched specimens, it is necessary to consider the influence of the notch angle, notch depth, and root radius on the yield stress, ultimate stress, breaking stress, and ductility. Attention will be given first to the influence of notches on the yield stress and ultimate stress. These indices of strength, when based on the minimum section, tend to increase with increase in the radial stress ratio (S_3/S_1) and to decrease with increase in the stress concentration induced by a notch. Both the radial stress ratio and the theoretical stress concentration tend to increase with decrease in the notch angle and root radius. With increase in the notch depth, however, the radial stress ratio tends to increase continuously, whereas the theoretical stress concentration increases to a maximum (reached at slight notch depth) and then decreases at an increasing rate. With

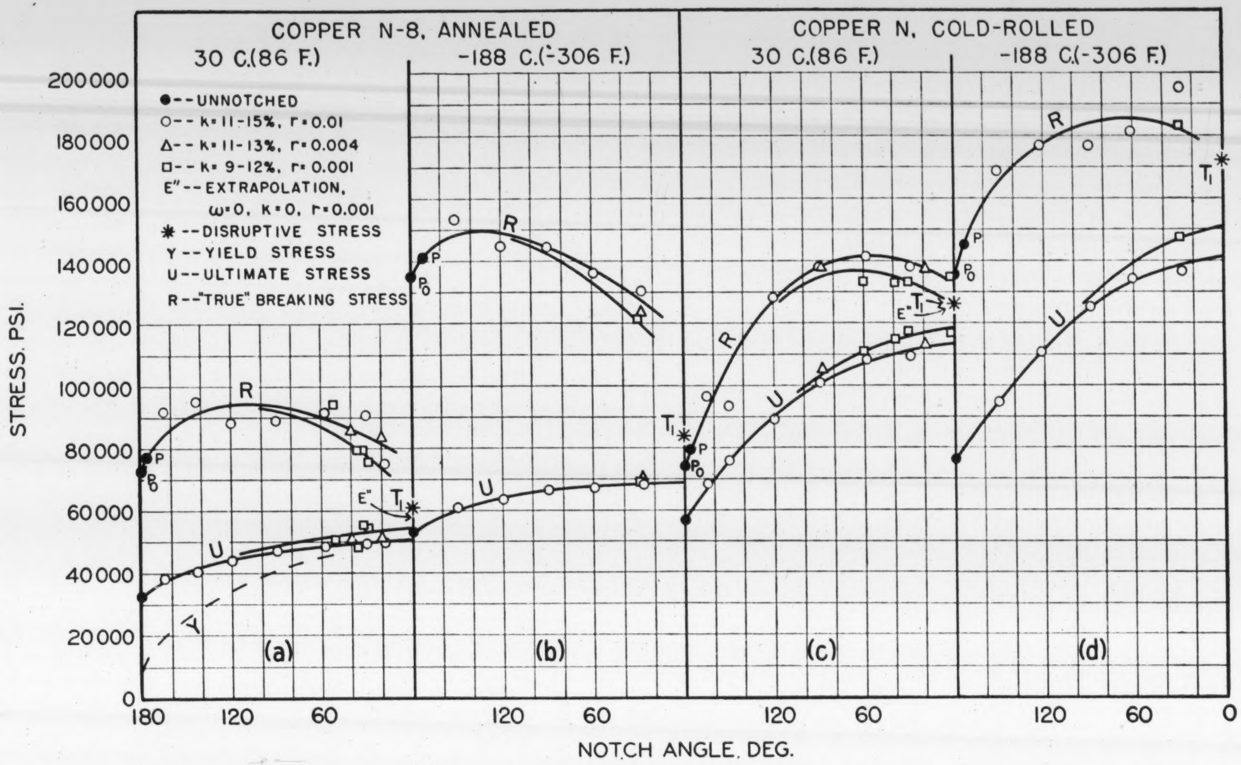

FIG. 1.—Influence of the Notch Angle on the Strength of Copper Specimens at Two Widely Different Temperatures.

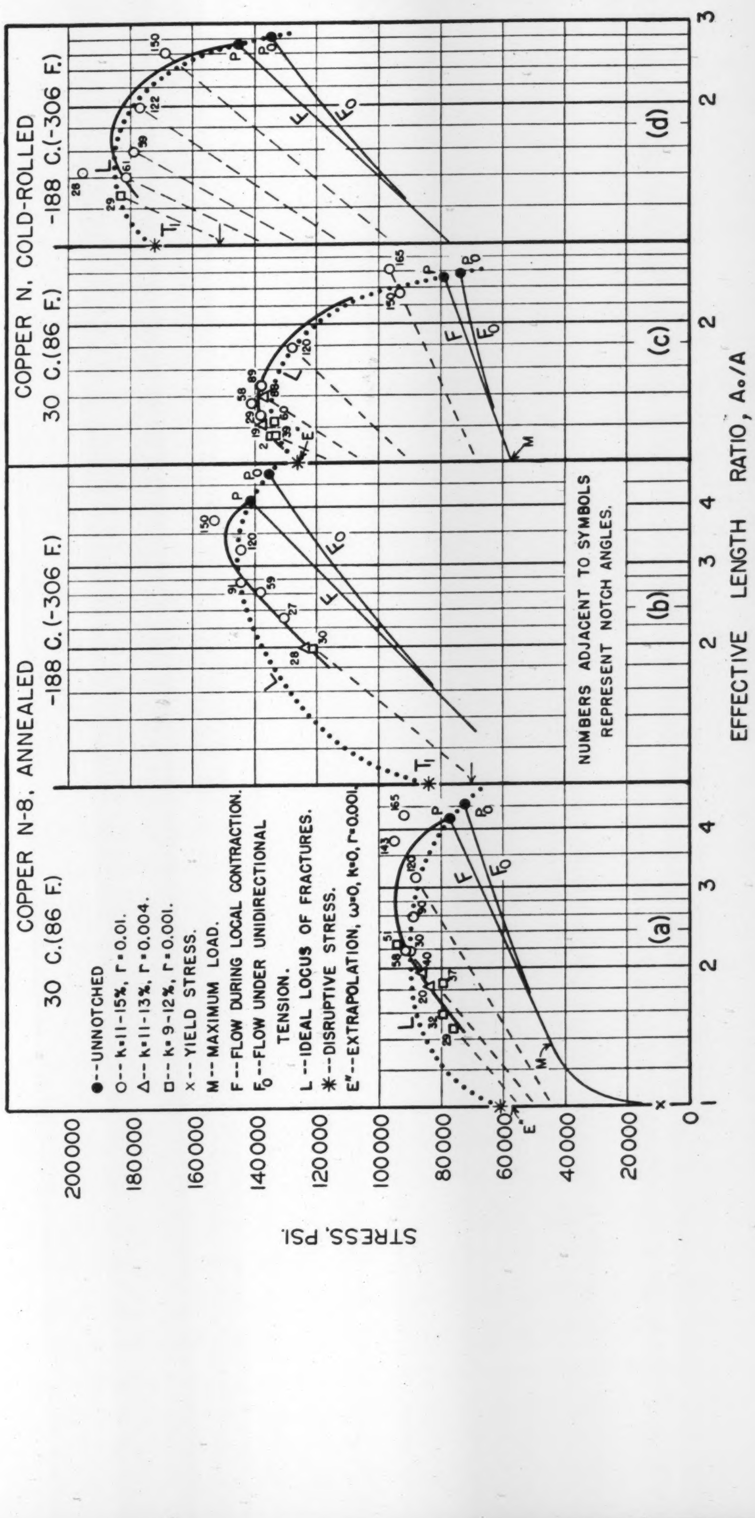

Fig. 2.—Influence of Notches on the Relation Between Breaking Stress and Ductility of Copper at Two Widely Different Temperatures.

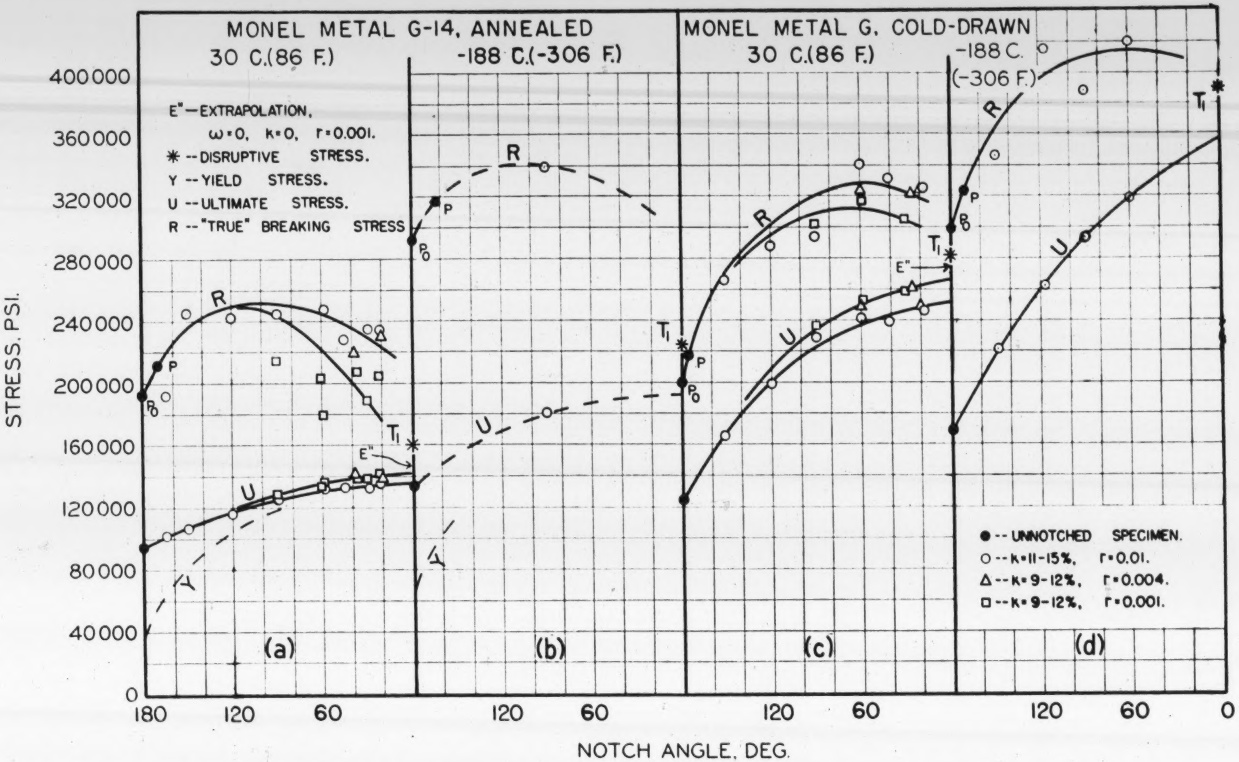

FIG. 3.—Influence of the Notch Angle on the Strength of Monel Metal Specimens at Two Widely Different Temperatures.

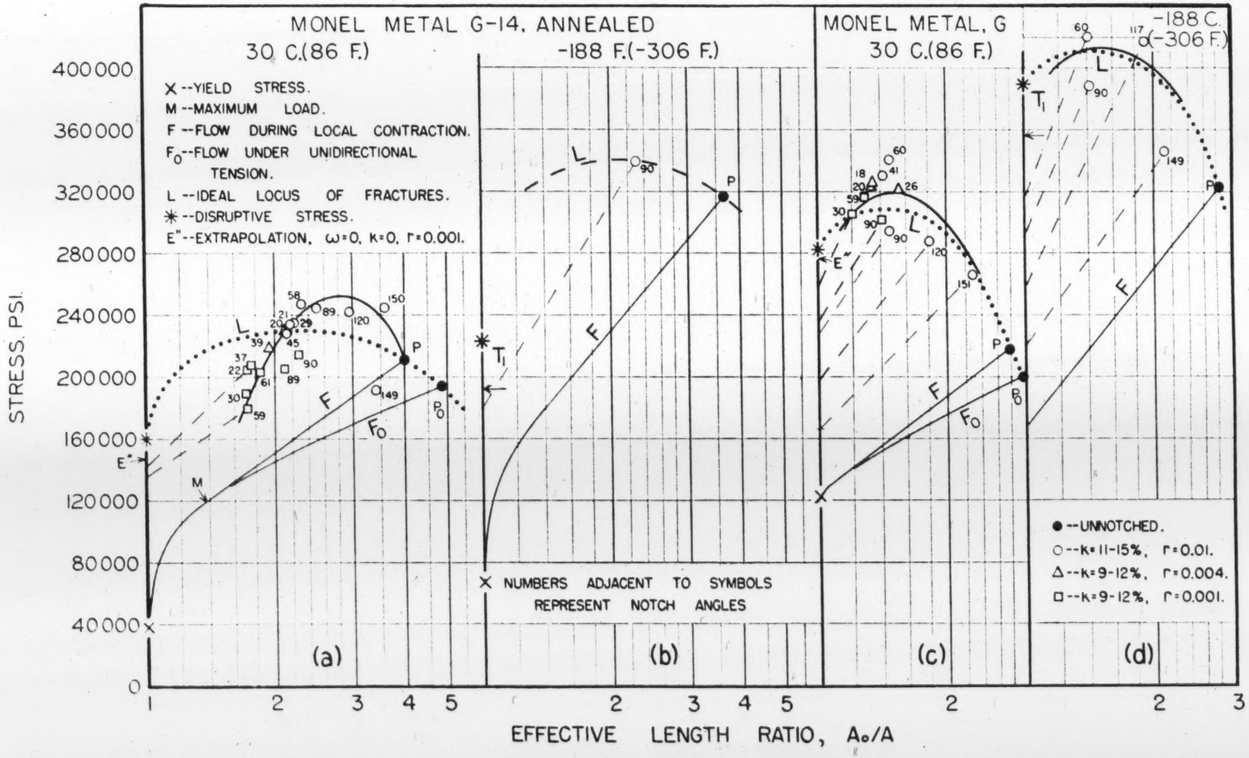

FIG. 4.—Influence of Notches on the Relation Between Breaking Stress and Ductility of Monel Metal at Two Widely Different Temperatures.

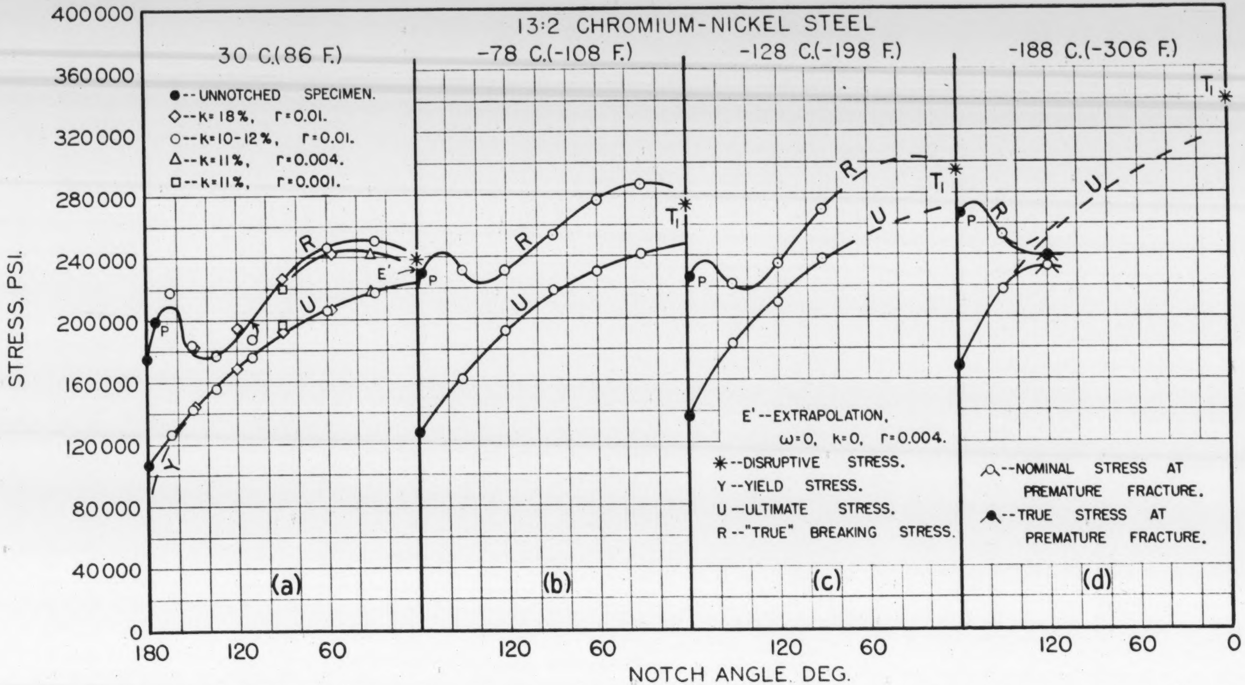

Fig. 5.—Influence of the Notch Angle on the Strength of Specimens of 13 per cent Chromium, 2 per cent Nickel Steel at Four Different Temperatures.

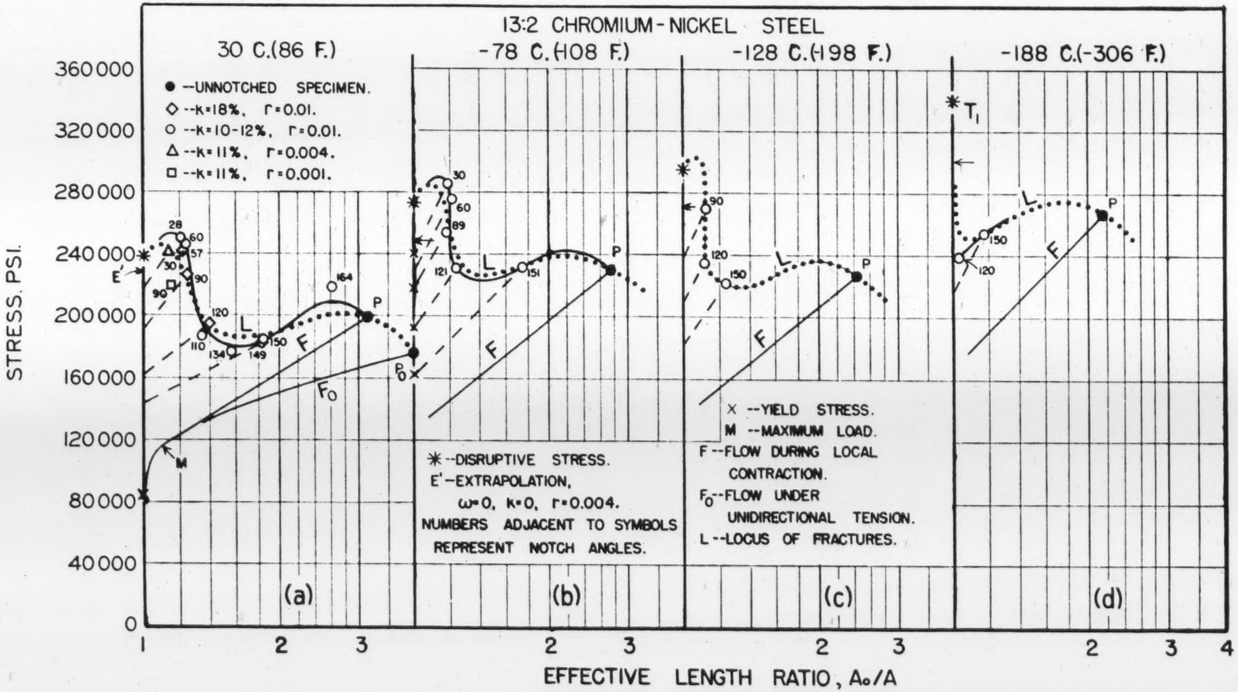

FIG. 6.—Influence of Notches on the Relation Between Breaking Stress and Ductility of 13 per cent Chromium, 2 per cent Nickel Steel at Four Different Temperatures.

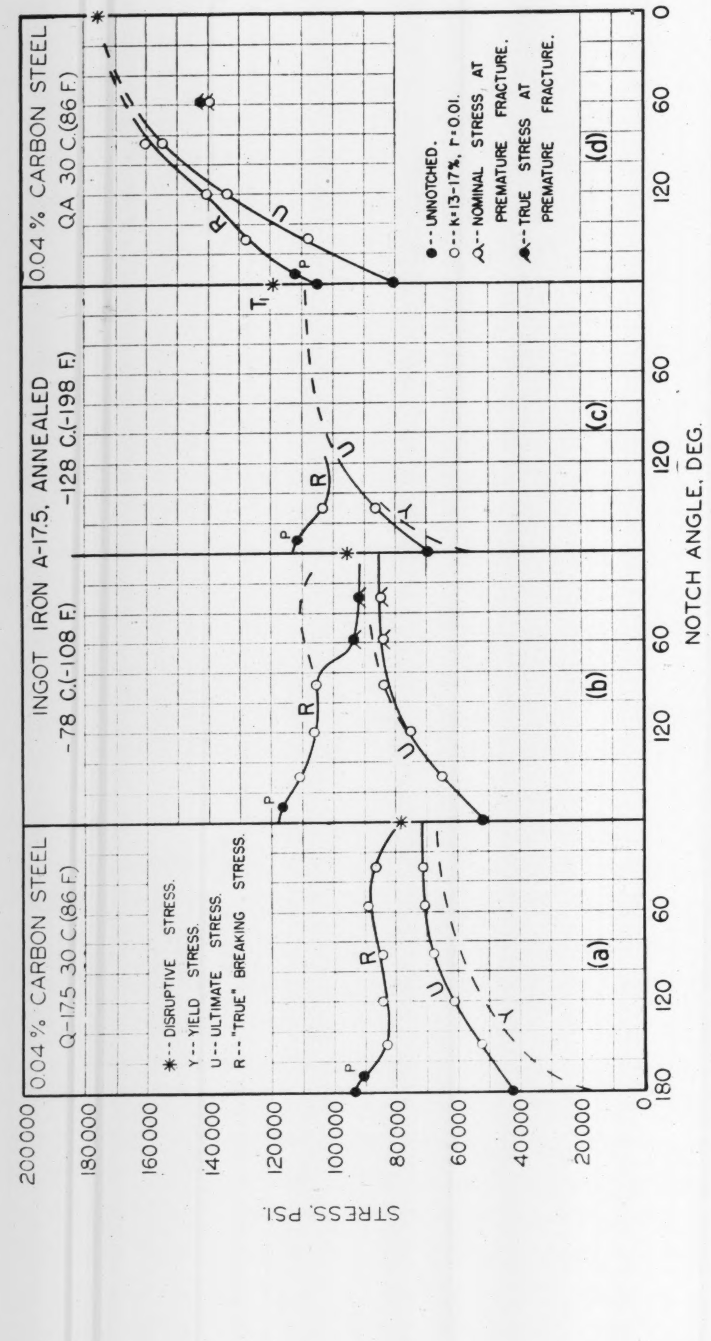

Fig. 7.—Influence of the Notch Angle on the Strength of Specimens of Low Carbon Steel at Different Temperatures.

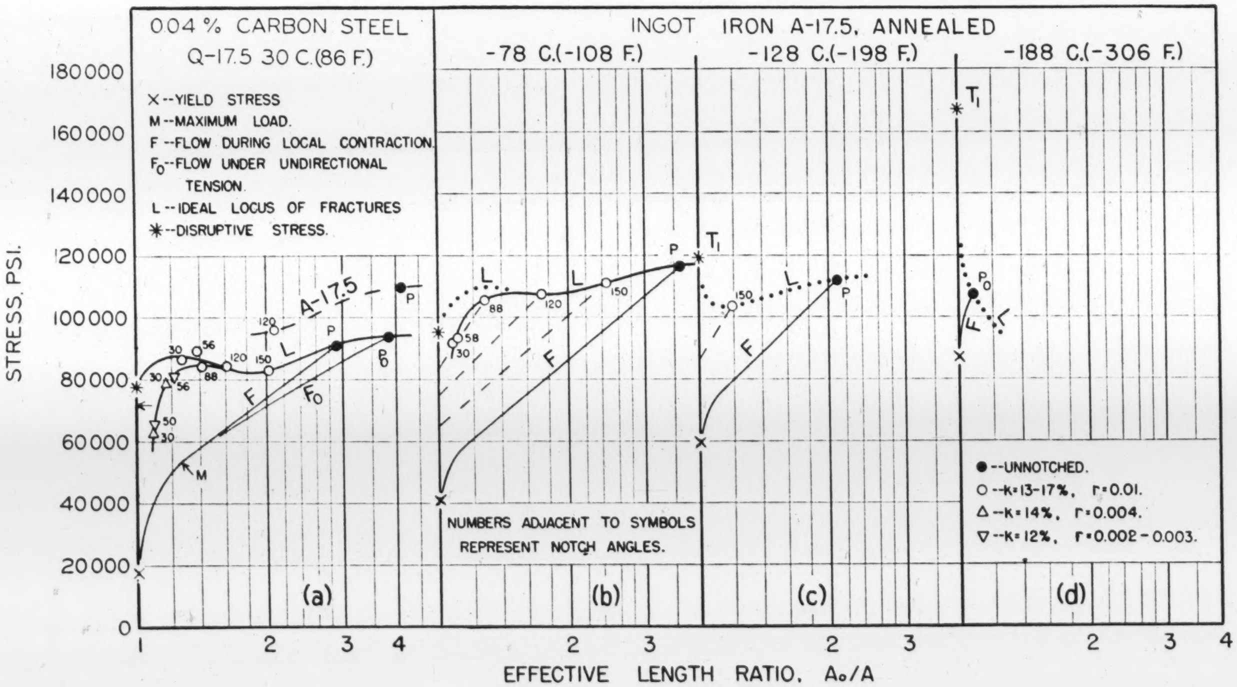

FIG. 8.—Influence of Notches on the Relation Between Breaking Stress and Ductility of Low Carbon Steel at Four Different Temperatures.

deeply notched specimens, consequently, the radial stress ratio becomes the dominant factor. As in the preceding papers (**11, 12, 13, 14**) the notch depths are represented indirectly in terms of the ratio, *k*, of the minimum to the maximum cross-sectional area. The deeper the notch, the smaller is the value of *k*. The specimens were all deeply notched, so that the *range* of values of *k* (indicated in each diagram) was as small as possible ($k = 9$ to 12 per cent). For nearly all the specimens, the notch radius, *r*, was 0.01 in.; for a few specimens, the radius was either 0.004 or 0.001 in.

Figures 1, 3, 5, and 7 show the influence of the notch angle, ω, on the yield stress, ultimate stress, and breaking stress, for ferrous and non-ferrous metals at room temperature and at the indicated low temperatures. Attention will be given first to the curves of yield stress and ultimate stress. With decrease in the notch angle from 180 deg. (unnotched specimen), these curves rise at a decreasing rate. The course of the curve is influenced somewhat by the notch radius. A curve obtained with notch radius 0.001 in. generally is considerably higher than the curves obtained with notch radius either 0.01 or 0.004 in. The rise of all these curves is due to an increase in the radial stress ratio (S_3/S_1). A qualitatively similar curve of ultimate stress is obtained when the notch angle, ω, is held constant and the notch depth is varied, except that the curve is generally depressed somewhat at intermediate notch depths (high values of *k*) because of the influence of stress concentration still present in shallow-notched specimens at maximum load (**14**). By curvilinear extrapolation, using the curves of variation of ultimate stress with the notch angle (Figs. 1, 3, 5, and 7) and the corresponding curves of variation of the ultimate stress with *k* (**14**), values may be obtained of the ultimate stress corresponding to zero values of ω and *k*. Values so obtained for the metals at room temperature are indicated by the points designated *E*, *E′*, or *E″* for values obtained with notch radii 0.01, 0.004, and 0.001 in., respectively. Values obtained by extrapolation from the curves of ultimate stress in Figs. 1, 3, 5, and 7 are indicated by horizontal arrows in some other figures to be discussed later.

The Influence of the Notch Angle and Root Radius on the Breaking Stress and Ductility:

The curves of breaking stress (Figs. 1, 3, 5, and 7), unlike the curves of ultimate stress, do not rise continuously with decrease in the notch angle. As shown in a preceding paper (**14**), the curves of breaking stress are of two types. The curves for copper and monel metal (Figs. 1 and 3) have a single rise and descent, with no reversal of curvature; the curves for ferritic steels have a reversal (Figs. 5 and 7). The course of a curve of breaking stress, in the absence of appreciable stress concentration at fracture, is the resultant of the influences of two factors on the technical cohesion limit. These factors are: The continuous increase of the radial stress ratio with decrease in the notch angle, and the continuous decrease of the total deformation at fracture (shown in Figs. 2, 4, 6, and 8, to be discussed later). A rise of a curve of breaking stress with decrease in the notch angle is due to dominance of the first factor; a descent is due to a dominance of the second factor, the continuous decrease in ductility.[5] The breaking-stress curves obtained with 0.004 and 0.001 in. notch radius, unlike the corresponding curves of ultimate stress, generally are below the corresponding curves obtained with notch radius 0.01 in.

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[5] As shown in the previous papers, plastic extension causes continuous increase in the technical cohesive strength, and hence *tends* to cause increase in any specific technical cohesion limit.

Opposite each of Figs. 1, 3, 5, and 7 are corresponding Figs. 2, 4, 6, and 8 showing the relation between breaking stress and ductility. Ductility is represented on a logarithmic scale, in accordance with the method described by MacGregor; abscissas represent logarithms of A_0/A_1, the ratio of the initial to the current cross-sectional area (**14**). On such a scale, equal differences in abscissa represent equal differences in nominal ductility. A line representing the variation of the flow stress[6] during local contraction is straight between the points representing maximum load and fracture. When the deformation at maximum load is not known, the course of such a line may be established by drawing a straight line between the point representing fracture and a point at the left representing the ultimate stress (nominal stress at maximum load). This method was used in constructing the flow stress lines obtained with notched specimens.

Each solid-line curve shows the influence of the notch angle on the relation between the breaking stress and nominal ductility. With decrease in the notch angle, the curves eventually turn downward. If such a curve represented a quantitatively correct relation between breaking stress and ductility, however, its intersection with the axis of abscissas ($A_0/A = 1$) would be above the point (E, E', or E'') representing the highest extrapolated value of the ultimate stress ($\omega = 0$, $k = 0$). The solid curves, however, generally turn downward much more rapidly than they would if abscissas represented true values of ductility. The curves for annealed copper and annealed monel metal (Figs. 2 and 4), if further extended, would even drop below the horizontal arrow representing the uppermost point on the U curve ($\omega = 0$) in the corresponding diagram of Figs. 1, 3, 5, or 7. As shown in a previous paper (**14**), however, the nominal ductility generally differs from the true ductility. Because there is a radial gradient of the degree of plastic deformation in the reduced section at fracture, the actual reduction of area at the point of beginning fracture cannot be measured. When fracture (as usual) starts near the specimen axis, the true ductility is less than the nominal ductility.

The dotted curves L represent qualitatively the approximate relation between breaking stress and true ductility. The vertical intercept, indicated by T_1, represents approximately the disruptive stress, the stress that would cause fracture under polarsymmetric tension ($S_1 = S_2 = S_3$). Under such a stress combination, a metal would fracture without prior plastic deformation, because there is no shearing stress. In establishing T_1, consideration must be given to the variation of the ultimate stress, breaking stress, and ductility with decrease in the notch angle. Point T_1 must be above any point representing the extrapolated value of the ultimate stress corresponding to zero values of ω and k. For details of the method of establishing approximately the position of T_1, reference may be made to one of the preceding papers (**14**).

The Influence of the Temperature of the Metal on the Form, Size, and Position of the Strength Diagram:

Consideration must now be given to the influence of the temperature of the metal on the form, size, and position of the previously described strength diagrams. In such a study, frequent com-

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[6] By "flow stress" is meant the yield stress based on the corresponding sectional area, not on the original area.

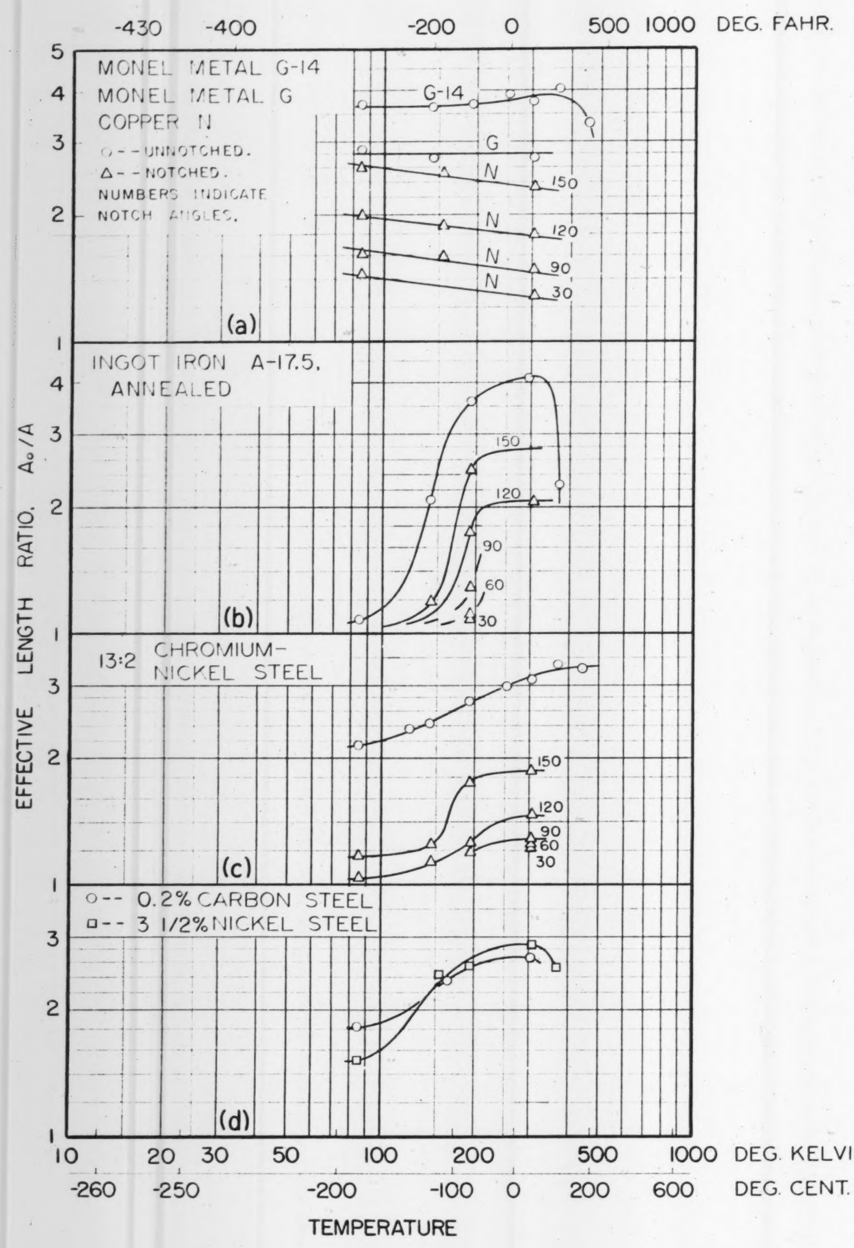

Fig. 9.—Influence of Temperature on the Ductility of Notched and Unnotched Specimens of Various Metals.

parison should be made between the correlated diagrams with abscissas representing notch angle and ductility, respectively.

In Figs. 1 to 4, comparison of diagrams (*a*) and (*c*) with diagrams (*b*) and (*d*), respectively, shows the effect of low temperature on the diagrams for copper and monel metal. Comparison of diagrams (*a*) and (*b*) with diagrams (*c*) and (*d*) shows the influence of prior plastic deformation. Lowering the temperature from 30 C. to −188 C. has had no effect on the qualitative forms of the curves, but has elevated the diagrams and increased the separation between the curves of ultimate stress and fracture. In Figs. 1 and 3, the curves of breaking stress have been elevated more than the curves of ultimate stress. In Figs. 2 and 4, lowering the temperature has elevated P and P_0 relatively more than T_1, and has thus tilted the entire diagram without appreciable change of form. The reason for this tilting of the diagram is revealed by comparison of the curves of flow stress, F, in diagrams (*a*) and (*c*) with those in diagrams (*b*) and (*d*) respectively. Lowering the temperature to −188 C. has greatly increased the slope of curve F. This increase in slope is due largely to the fact that the hardening during plastic deformation is greater at the lower temperature. Although the *amount* of plastic deformation at fracture of copper and monel metal differs little at the two temperatures, the strengthening effect of the deformation is much greater at −188 C. than at room temperature. The tilting of the strength diagram (Figs. 2 and 4) due to the lowering of the temperature is greater for an annealed metal than for a previously cold-worked metal because the work hardening of the annealed metal is all done at −188 C.

The technical cohesive strength of the ferritic steels was investigated at 30 C., −78 C., −128 C., and −188 C. However, because of the decrease in ductility of these steels as the temperature is lowered (diagrams (*c*) and (*d*) of Fig. 9), only incomplete diagrams of cohesive strength were obtained at −128 C. and −188 C. (Figs. 5 to 8). With the 13 per cent chromium, 2 per cent nickel steel at −128 C. (Figs. 5 (*c*) and 6 (*c*)), the ductility was sufficient to permit the load to reach a true maximum when the notch angle was 90 deg. or more. At −188 C., however, fracture was definitely premature when the notch angle was 120 deg. (Figs. 5 (*d*) and 6 (*d*)). With the ingot iron at −128 C. (Figs. 7(*c*) and 8(*c*)) the load barely reached a true maximum when the notch angle was 150 deg. At −188 C., the ductility was barely enough to permit the determination of the ultimate stress with an unnotched specimen. Even at −78 C., fracture was premature when the notch angles were 58 and 30 deg.

The premature fractures of notched specimens were obtained because the ductility was insufficient to permit relief of stress concentration before the load reached a maximum. If the desired radial stress ratios could be obtained without nonuniform stress distribution, it would be possible to obtain a complete diagram even with a metal that is brittle under any stress system. Broken lines are used in Figs. 5 to 8 to indicate the approximate form of such diagrams at −128 and −188 C. The dotted lines, L, represent the approximate course of the locus of fractures if abscissas represented values of true ductility.

The evidence in Figs. 1 to 8 indicates that a decrease of temperature within the range from 30 to −188 C. does not change the form of a diagram of technical cohesion. The effect of low temperature, in this respect, is different from the effect of work hardening. Comparison of diagrams (*a*) and (*d*) of Fig. 7 for

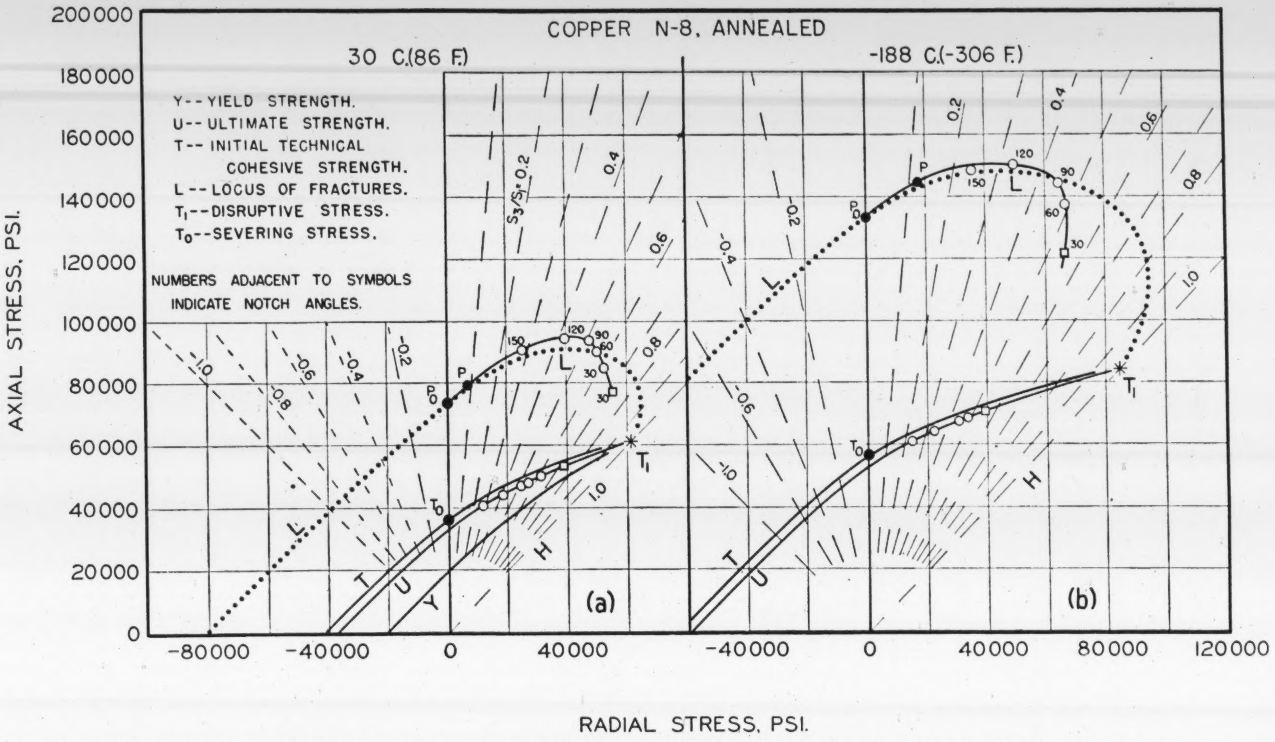

FIG. 10.—Influence of the Radial Stress Ratio on the Yield Stress, Ultimate Stress, and Technical Cohesion Limit of Annealed Copper at Two Widely Different Temperatures.

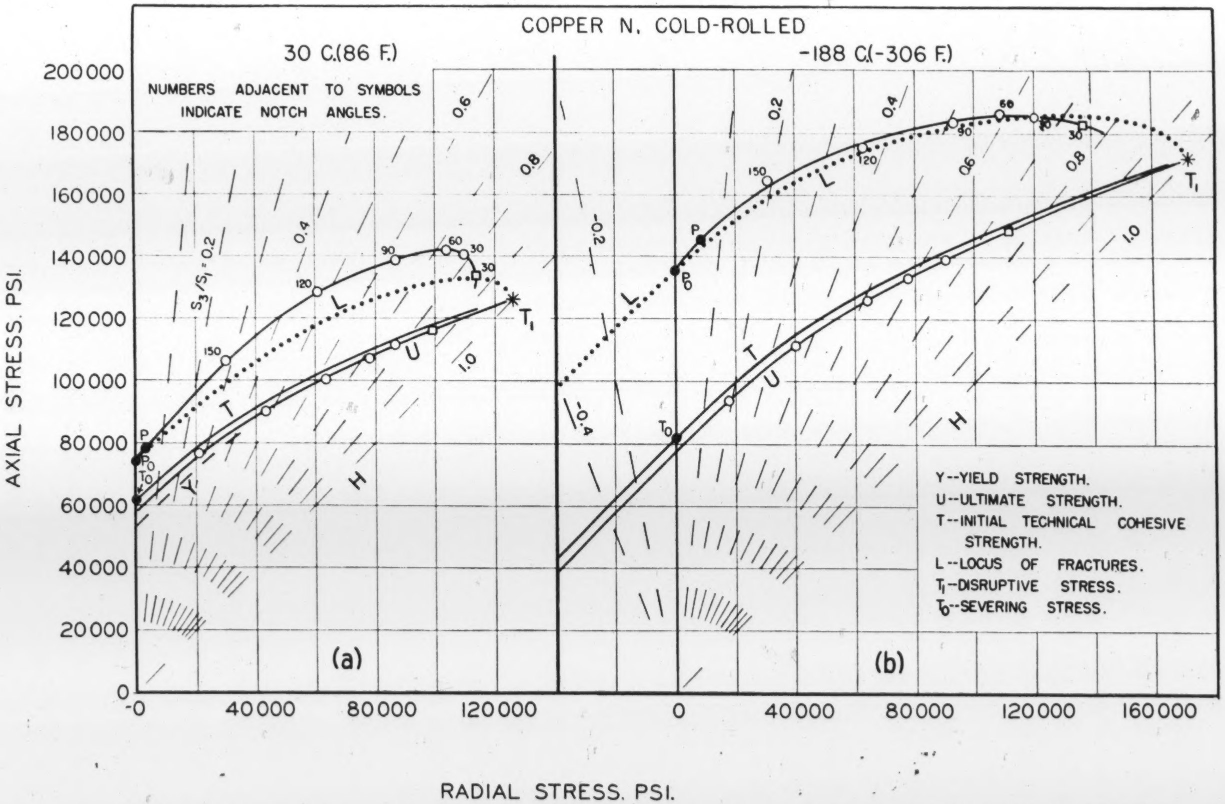

FIG. 11.—Influence of the Radial Stress Ratio on the Yield Stress, Ultimate Stress, and Technical Cohesion Limit of Cold-Drawn Copper at Two Widely Different Temperatures.

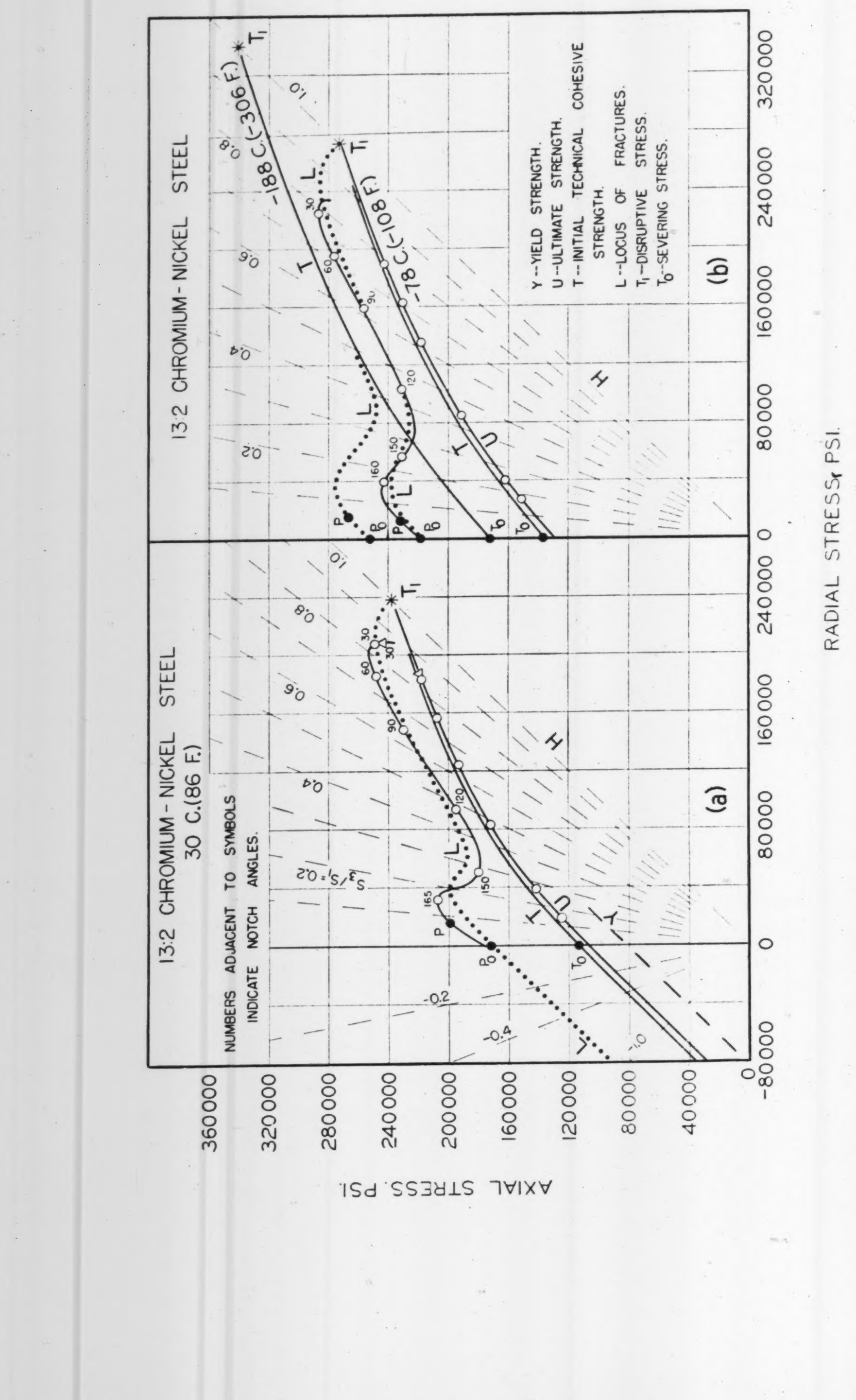

FIG. 12.—Influence of the Radial Stress Ratio on the Ultimate Stress and Technical Cohesion Limit of 13 per cent Chromium, 2 per cent Nickel Steel at Three Different Temperatures.

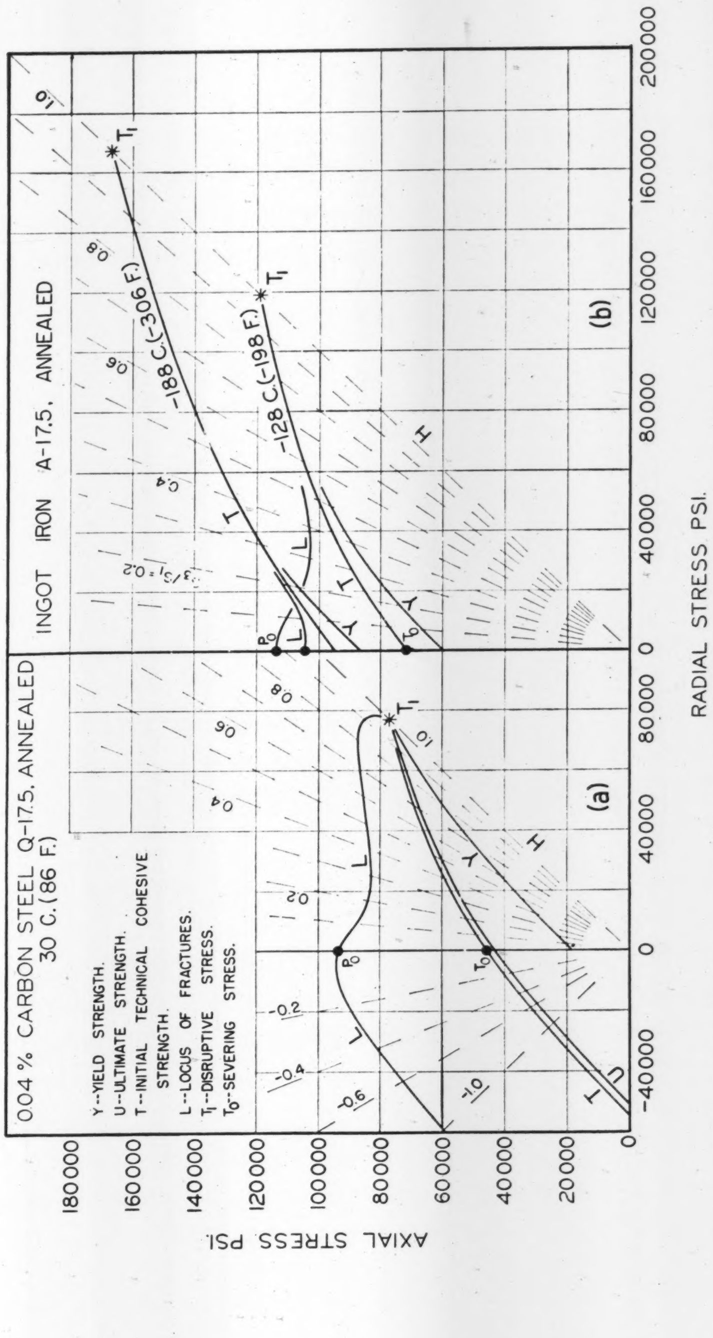

FIG. 13.—Influence of the Radial Stress Ratio on the Yield Stress, Ultimate Stress, and Technical Cohesion Limit of Low Carbon Steel at Three Different Temperatures.

annealed and cold-drawn steels, respectively, shows that prior plastic deformation not only elevates the diagram for the 0.04 per cent carbon steel but changes its shape.

For a detailed study of the influence of low temperature on ductility, comparison may be made between the positions of corresponding experimental points in diagrams obtained at different temperatures. In the diagrams for copper (Fig. 2), points representing corresponding notch angles generally indicate slightly greater ductility at −188 C. than at +30 C. In the diagrams for monel metal (Fig. 4) the points representing corresponding notch angles indicate about the same ductility at these two temperatures. A very different relationship, however, is found in the diagrams for the ferritic steels (Figs. 6 and 8). The ductility of these steels decreases considerably as the temperature is lowered. This effect of lowering the temperature increases with decrease in the notch angle.[7]

The quantitative influence of temperature on the ductility of these two classes of metal is shown in Fig. 9. Whereas the ductility of the copper and monel metal shows little change with the lowering of the temperature, the ductility of the two ferritic steels decreases greatly. With some notch angles, the ductility decreases abruptly within a narrow range of temperature. The smaller the notch angle, the higher is the mean temperature of the most rapid decrease in ductility. The relations between temperature and ductility evidently are qualitatively similar to the relations between temperature and resistance to impact (**10**).

The Influence of the Combination of Principal Stresses on the Technical Cohesion Limit, Yield Stress, and Ultimate Stress at Different Temperatures:

From the diagrams in Figs. 1 to 8 other diagrams may be derived to represent the influence of the combination of principal stresses on the technical cohesion limit, yield stress, and ultimate stress. Such diagrams are shown in Figs. 10 to 13. In these diagrams, abscissas represent radial stresses, and ordinates represent axial stresses. Each of the radiating broken lines is the locus of points representing the indicated constant value of the radial stress ratio; the diagonal line *H* is the locus of points representing polarsymmetric stress ($S_1 = S_2 = S_3$). Points representing results of tension tests of notched specimens are in the field of positive values of S_3/S_1.

The method of deriving these diagrams is described in a previous paper (**14**). As actual values of the radial stresses obtained with the notched specimens are not known, the diagrams must be considered merely qualitative representations based on certain empirical assumptions. One of the assumptions is that the radial stress ratio varies linearly with the ultimate stress, from zero for an unnotched specimen to a value of 1.0 when the ultimate stress equals the disruptive stress. This assumption is the basis for the location of the small circles traversed by curves *U* (Figs. 10, 11, and 12) which are derived from corresponding positions on curves *U* in Figs. 1, 3, and 5. It is also assumed that the radial stress ratio does not change during the plastic deformation between maximum load and fracture.[8] This assumption leads to the location of the small circles traversed by the solid curves of breaking stresses (Figs. 10, 11, and 12), which are derived

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[7] In Fig. 8, allowance should be made for the fact that the ductility of the 0.04 per cent carbon steel is considerably less than that of the ingot iron.

[8] The actual change of S_3/S_1 during this local contraction is the resultant of three factors, the changes in the notch depth, notch angle, and root radius (14).

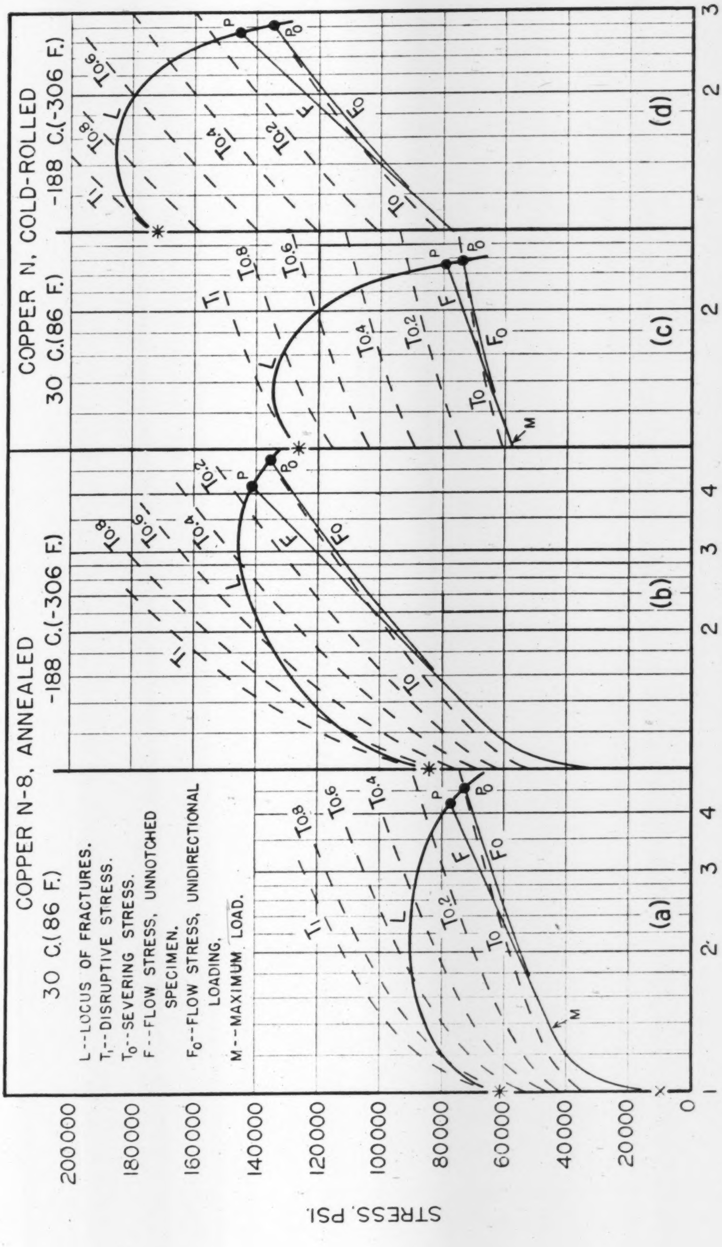

Fig. 14.—Influence of Plastic Deformation on the Technical Cohesive Strength of Copper at Two Widely Different Temperatures.

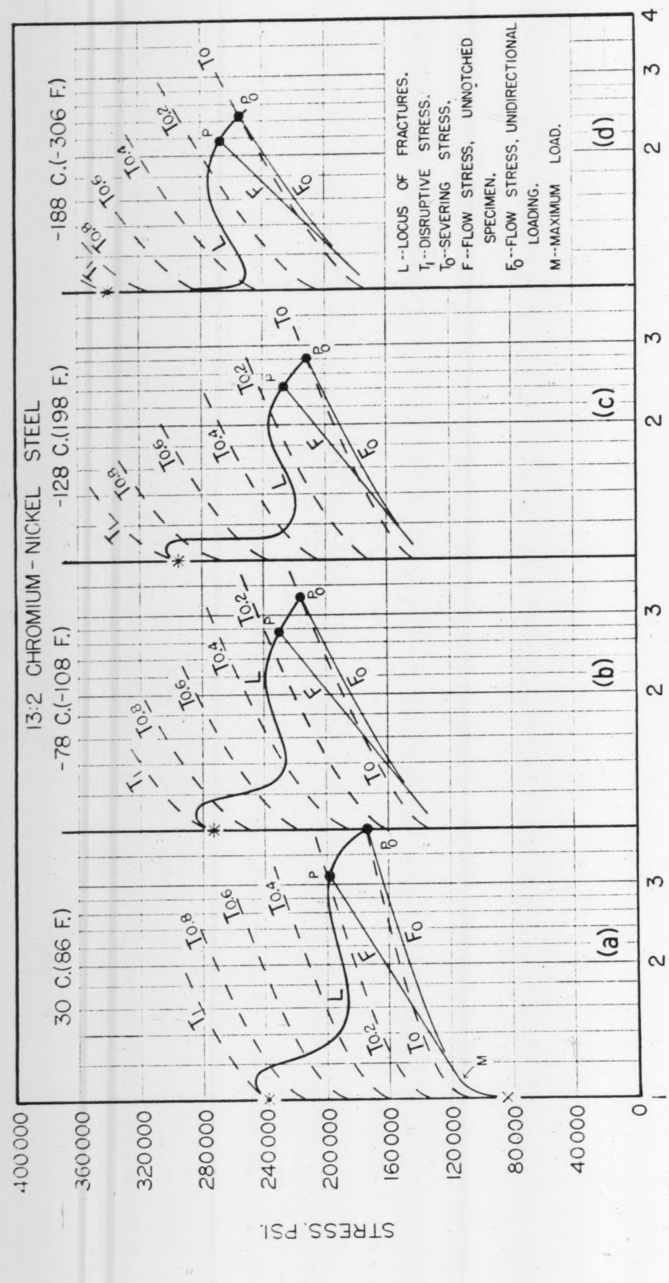

Fig. 15.—Influence of Plastic Deformation on the Technical Cohesive Strength of 13 per cent Chromium, 2 per cent Nickel Steel at Four Different Temperatures.

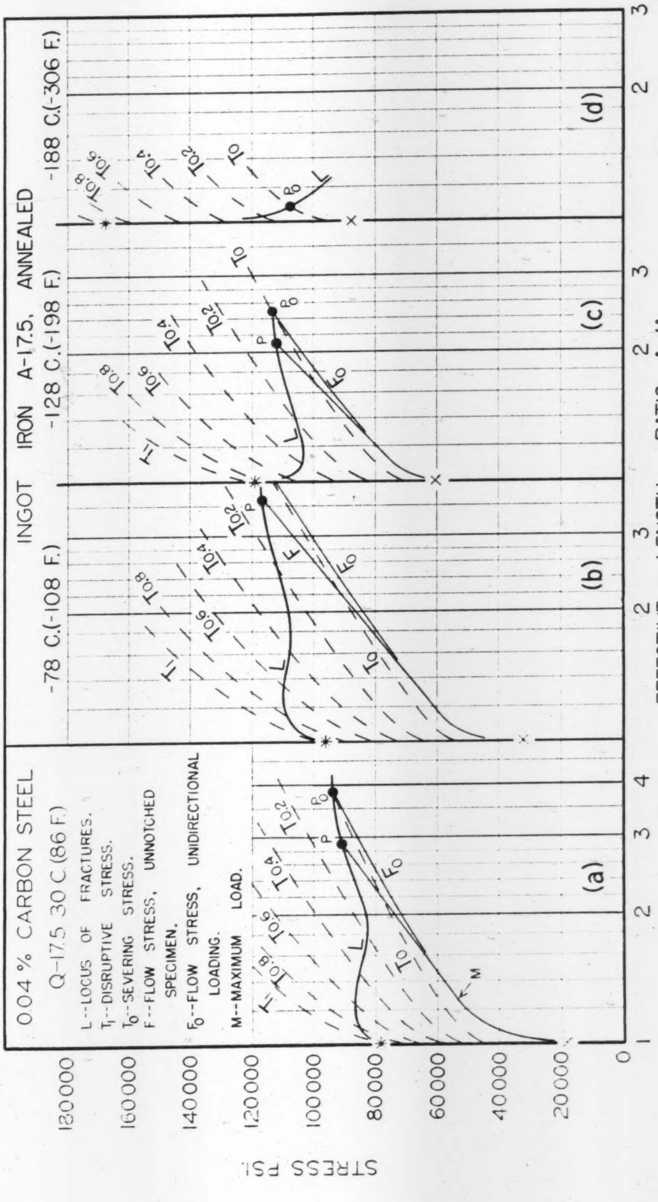

FIG. 16.—Influence of Plastic Deformation on the Technical Cohesive Strength of Low Carbon Steel at Four Different Temperatures.

from corresponding positions on curves R in Figs. 1, 3, and 5. For reasons given in the discussion of Figs. 2, 4, 6, and 8, the ideal locus of fractures in each of Figs. 10, 11, and 12 must reach line H at a point, T_1, which represents the disruptive stress, and which corresponds to point T_1 in the basic diagram (Figs. 2, 4, 6, or 8). The ideal locus of fractures is represented approximately by the curve L in each diagram of Figs. 10 to 13. This curve represents qualitatively the course that the locus of fractures would take if the specimens could be subjected to combinations of uniform axial and radial stresses.

Point T_1 in each diagram is the junction of the locus of fractures and the curves of yield strength, ultimate strength, and initial technical cohesive strength. Curve T represents approximately the initial technical cohesive strength. Point T_0 represents the technical cohesion limit of unaltered metal under unidirectional stress. Point P_0 represents fracture of the plastically deformed metal under unidirectional tension. The intersections of curve T with the radiating broken lines represent cohesion limits for the corresponding values of S_3/S_1. Each of such cohesion limits may be denoted by T with the corresponding value of S_3/S_1 as subscript (**11, 12, 13, 14**). Because plastic deformation raises the technical cohesive strength, fracture would not occur at stresses represented by any point on curve T except T_1, but would occur when the stress is raised to the corresponding point on curve L. The flow stress would then equal the technical cohesion limit for that stress system.

Decrease in the temperature from 30 C. to −188 C. elevates curve T and thus increases the area enclosed by that part of the diagram representing the initial technical cohesive strength. For copper (Figs. 10 and 11), the decrease in temperature raises curve L relatively more than curve T. This differential effect is due partly to the slightly increased ductility, but is largely due to the greater degree of work hardening at the lower temperature. Point P_0 is thus elevated with reference to T_1. For the ferritic steels (Figs. 12 and 13), the decrease in temperature elevates curve L less than curve T. This effect is due to the decrease in ductility, and this in turn is due to the fact that a decrease in temperature increases the technical cohesive strength of ferritic steels less than it increases the resistance to plastic deformation.

The locus of fractures, L, in the derived diagrams (Figs. 10 to 13) is of two forms, corresponding to the previously discussed two forms of the curves of breaking stresses in the basic diagrams. The curves L for copper and for monel metal (not shown) have no reversal of curvature, whereas the curves for the ferritic steels are more complex.

The Influence of Plastic Extension on the Technical Cohesive Strength of Metals at Different Temperatures:

Diagrams of another type, similar to those in Figs. 2, 4, 6, and 8, have been derived to represent the influence of plastic deformation on the technical cohesive strength of metals (**11, 12, 14**), and are shown in Figs. 14, 15, and 16. The locus of fractures in each of these derived diagrams is the same as the ideal locus of fractures in the corresponding basic diagram, discussed previously. The influence of plastic deformation on the technical cohesive strength of a metal cannot be represented by a single curve, but may be represented by a series of curves, each showing the variation of the cohesion limit for a specific radial stress ratio. Such a series is shown in each of the diagrams of Figs. 14, 15, and 16. The intersection of each of the broken lines with the locus of fractures represents fracture under the indicated stress system. Point P represents fracture of an initially unnotched specimen after local contraction. Point P_0 represents fracture under unidirectional stress, and curve F_0 represents the hypothetical course of the flow stress in the absence of the notch effect of the increasing local contraction. By repeated interruption of a tension test and machining to diminish this notch effect, the relations between curves F_0 and F, and points P_0 and P have been determined approximately (14).

The difference in form between the locus of fractures for ferritic steels and non-ferrous metals may be attributed to a difference in the differential effect of an increase in S_3/S_1 on corresponding curves of flow stress and cohesion. With increase in S_3/S_1 a curve of flow stress is elevated relatively to the corresponding curve of cohesion and thus the ductility is decreased. This differential effect evidently is greater for the ferritic steels than for the non-ferrous metals. The abrupt decrease in ductility of the ferritic steels within a certain range of S_3/S_1 (Figs. 14, 15, and 16), and the abrupt decrease in ductility of notched specimens of these steels within a certain range of temperature (diagrams (*b*) and (*c*) of Fig. 9) may be correlated with the steep drop in impact value of these steels within a narrow range of temperature. The position of this temperature range depends on the composition, on the form and size of the specimen, and on the velocity of deformation. The greater the velocity the higher is the temperature of the steep drop in impact value (10). This variation of ductility, however, does not imply that an increase in velocity raises the flow stress more than it raises the technical cohesive strength, or that an increase in velocity decreases the cohesive strength. An increase in velocity may merely give less time for redistribution of stress, and thus may result in a larger value of S_3/S_1, more stress concentration, and a greater tendency to "premature" fracture.

THE VARIATION OF THE STRENGTH OF METALS BETWEEN ROOM TEMPERATURE AND −188 C.

Previous Investigations:

The literature gives no consistent picture of the variation of the strength of metals between room temperature and that of liquid air. The authors know of only two investigations of the tensile properties of metals at a temperature below that of liquid air. F. A. Lindemann and C. L. Lindemann (9) gave results of tests of various metallic elements at room temperature and at the temperatures of liquid air and liquid hydrogen. As they used wires about 0.05 mm. in diameter, not accurately measured and of uncertain composition and treatment, their results have little quantitative significance. The majority of the results, however, indicate considerably greater tensile strength at −252.6 C. than at −188 C. DeHaas and Hadfield (4) investigated the strength of metals at the temperature of liquid hydrogen. They found very little difference between the ultimate stress values obtained at this temperature and the values obtained by Hadfield (6) in a much earlier investigation with some of the same metals at the temperature of liquid air. They thus came to the conclusion that the strength of metals reaches an approximate limit at the temperature of liquid air. No comparison was made between yield stresses or breaking stresses at the two temperatures. As shown by Hadfield (6) and by others (7, 8, 21, 22), the strength of metals is much greater at −188 C. than at room temperature. Their results, to-

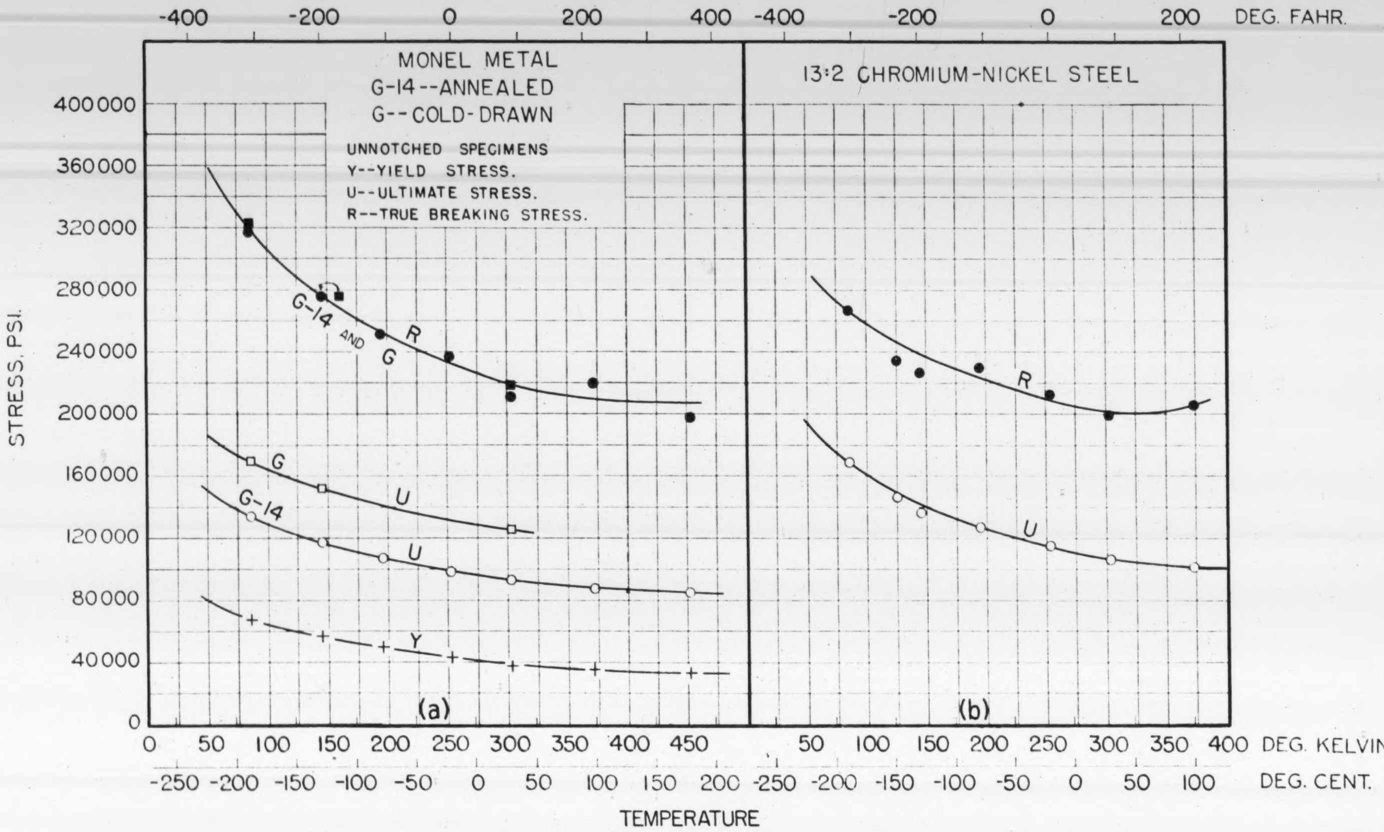

FIG. 17.—Variation of the Strength of Monel Metal and 13 per cent Chromium, 2 per cent Nickel Steel with Temperature Represented on the Usual Arithmetical Scale.

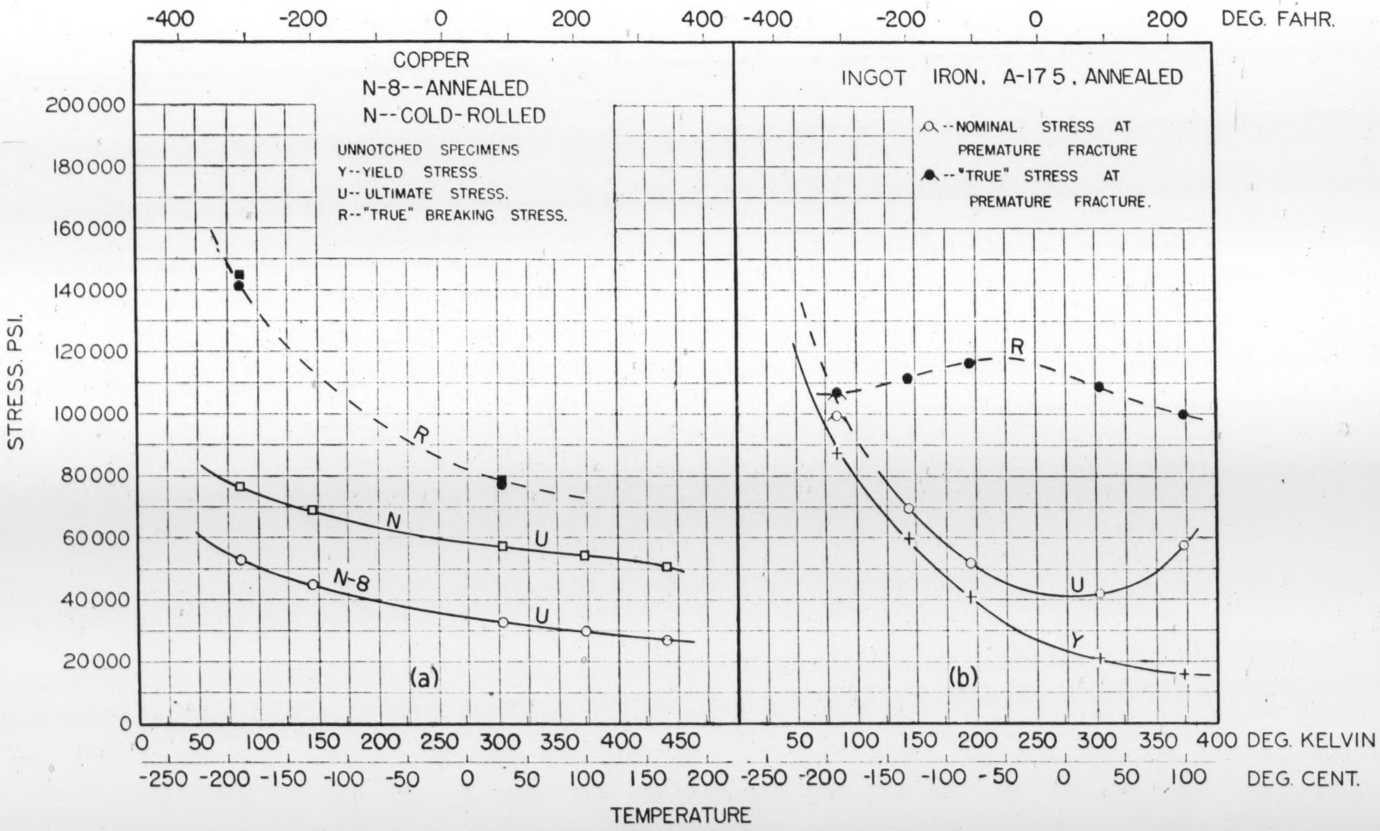

Fig. 18.—Variation of the Strength of Copper and Ingot Iron with Temperature Represented on the Usual Arithmetical Scale.

gether with those obtained by other investigators at the temperature of solid CO_2 (−78 C.) imply that the strength of metals rises very rapidly between −78 C. and −188 C. If the strength rises rapidly within this range but increases no more with further decrease of temperature, there must be either an abrupt reversal of the strength-temperature curve or this curve must end at the temperature of liquid air.

Results obtained by Inokuti (7) and by Broniewski and Wesolowski (3), however, indicate that the strength of metals rises at an increasing rate as the temperature is lowered to −188 C. Results of an investigation by Fahrenhorst and Schmid (5) of the resolved critical shearing stress of single crystals indicate a similar increase of strength, possibly continuing down to the temperature of liquid hydrogen (−252 C.). This evidence, however, is inconclusive because of the wide scatter of results.

The Variation of the Strength of Metals with Temperature Represented on the Usual Scale:

The following study of the variation of the strength of metals with temperature is based not only on the data previously discussed in connection with Figs. 1 to 16 but on considerable additional data. In addition to the metals previously discussed, experiments have also been made with nickel, aluminum, copper-tin alloy, 0.2 per cent carbon steel, and a steel containing 0.30 per cent carbon and 3½ per cent nickel. Experiments have been made at temperatures so distributed as to show conclusively the variation of the strength of the metals with temperature. Results obtained with some of the metals are shown in Figs. 17 and 18. Abscissas are on the Kelvin scale of temperatures, and corresponding positions on the Centigrade and Fahrenheit scales are indicated.

In Figs. 17 (*a*) and 18 (*a*) are curves of variation of the strength of monel metal and copper, respectively. The curves of yield stress, ultimate stress, and "true" breaking stress are all similar in form. The evidence indicates that, on this scale of temperature, the strength of these metals rises at an increasing rate from room temperature to −188 C. The curves give no suggestion that the strength values reach a limit at the temperature of liquid air. The ultimate-stress curve for 13 per cent chromium, 2 per cent nickel steel (Fig. 17 (*b*)) shows a similar rise throughout the same range of temperature. Similar curves (not shown) were obtained with nickel, aluminum, phosphor bronze (copper-tin alloy), 0.2 per cent carbon steel, and 3½ per cent nickel steel (S.A.E. 2330).

A similar variation, moreover, is found in the yield stress curve for ingot iron (Fig. 18 (*b*)) and in the ultimate stress curve between the minimum and −188 C. To the right of this minimum, the rise of the ultimate stress curve reveals the influence of age hardening during the tension test. This influence becomes appreciable somewhat below room temperature and is prominent at 100 C. The total effect depends greatly on the rate of plastic deformation during the test. The ultimate stress value at 100 C. was obtained by a test at a very slow rate.

The breaking stress curves for monel metal and 13 per cent chromium, 2 per cent nickel steel are similar in form to the curves of ultimate stress, except that the curve for the steel shows a change of trend above room temperature. This change may be attributed to the age-hardening tendency exhibited in varying degree by the ferritic steels. The entire breaking stress curves, however, are affected by other variables associated with the variation in temperature. One of these variables is ductility. When ductility decreases rapidly with decrease of temperature, as it does for ingot iron (Fig. 9), this variable becomes dominant and interrupts the rise of the curve of breaking stress (Fig. 18 (*b*)). In the breaking stress curves for monel metal and 13 per cent chromium, 2 per cent nickel steel, however, variable ductility does not become a dominant factor, and these curves show qualitatively the variation of the technical cohesion limit corresponding to a nearly constant low value of the radial stress ratio; they, therefore, show qualitatively the variation of the technical cohesive strength with temperature represented on the Kelvin scale.

Comparison Between the Variation of Strength and the Variation of Some Physical Properties with Temperature:

The continuously increasing slope of the curves of variation of strength with decrease of temperature (Figs. 17 and 18) has no counterpart in the curves of variation of physical properties in Fig. 24. The modulus of elasticity increases at a *decreasing* rate with decrease of temperature. With the exception of metals with low melting point, the modulus of elasticity increases very little between room temperature and −188 C.

Some physical properties, however, change greatly within this temperature range. The variations of the atomic heat and of the total heat content of some of the common metals are shown in Fig. 24.[9] A curve of variation of the coefficient of expansion would be similar in form to the curves of atomic heat. With decrease of temperature, each of these curves descends eventually at a *decreasing* rate, whereas the curves of variation of strength rise at an *increasing* rate. The only similarity between the curves representing strength and the curves representing the physical properties consists in the fact that the most rapid change of the property is within a temperature range far below room temperature.

The comparison of Fig. 24 with Figs. 17 and 18 suggests that both the resistance to plastic deformation and the technical cohesive strength of metals may have a thermodynamic basis and thus may be determined by values of energy rather than by values of force. However, there is another possible interpretation of the variation of strength with temperature.

The Variation of the Strength of Metals with Temperature Represented on a Scale in Which Equal Intervals Correspond to Equal Thermodynamic Efficiencies:[10]

The original thermodynamic scale of temperatures and the later one, now in general use, were both proposed by Kelvin (**23**). Both these scales are based on the principle of the Carnot cycle. According to the second law of thermodynamics $(Q_1 - Q_2)/Q_1 = (T_1 - T_2)/T_1$. In this equation, Q_1 is the heat added to a system at temperature T_1 (on Kelvin's second scale, now called the "Kelvin scale") and Q_2 is the heat removed at a lower temperature T_2. The term on the left of the equation represents the thermodynamic efficiency, the fraction of the transferred heat theoretically available as mechanical work. The equation may also be written in the form $\Delta Q/Q = \Delta T/T$. On Kelvin's original thermodynamic scale, equal temperature intervals correspond to equal thermodynamic efficiencies and hence correspond to equal values of $\Delta T/T$, that is, to equal values of $d \log T$. To represent tem-

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[9] For reasons that will become apparent later, temperature is here represented on a logarithmic scale. The curves would be qualitatively similar, however, if temperature were represented on the usual arithmetical scale.

[10] The opinions expressed in this and the following sections are those of the authors and not the National Bureau of Standards.

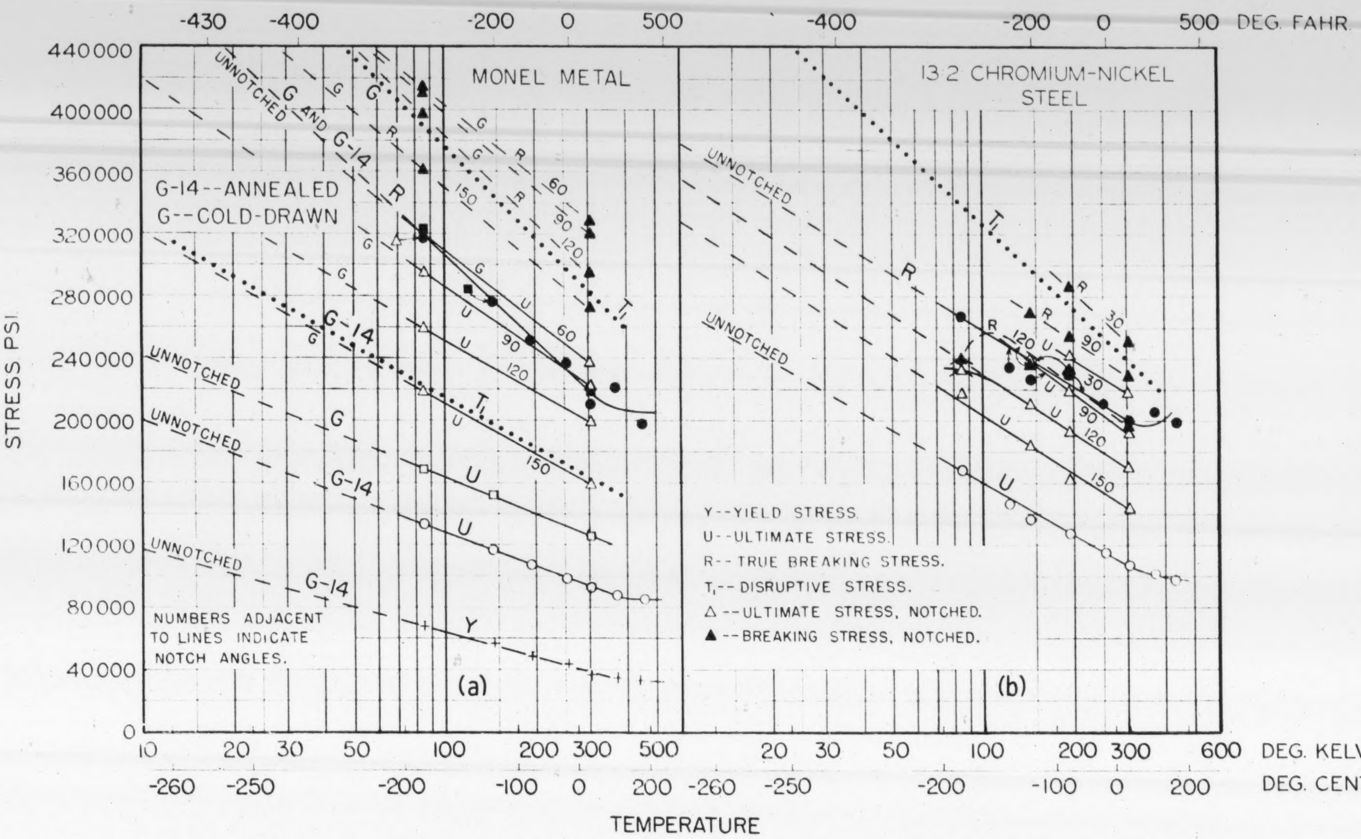

Fig. 19.—Variation of the Strength of Monel Metal and 13 per cent Chromium, 2 per cent Nickel Steel with Temperature Represented on a Scale of Equal Thermodynamic Efficiencies.

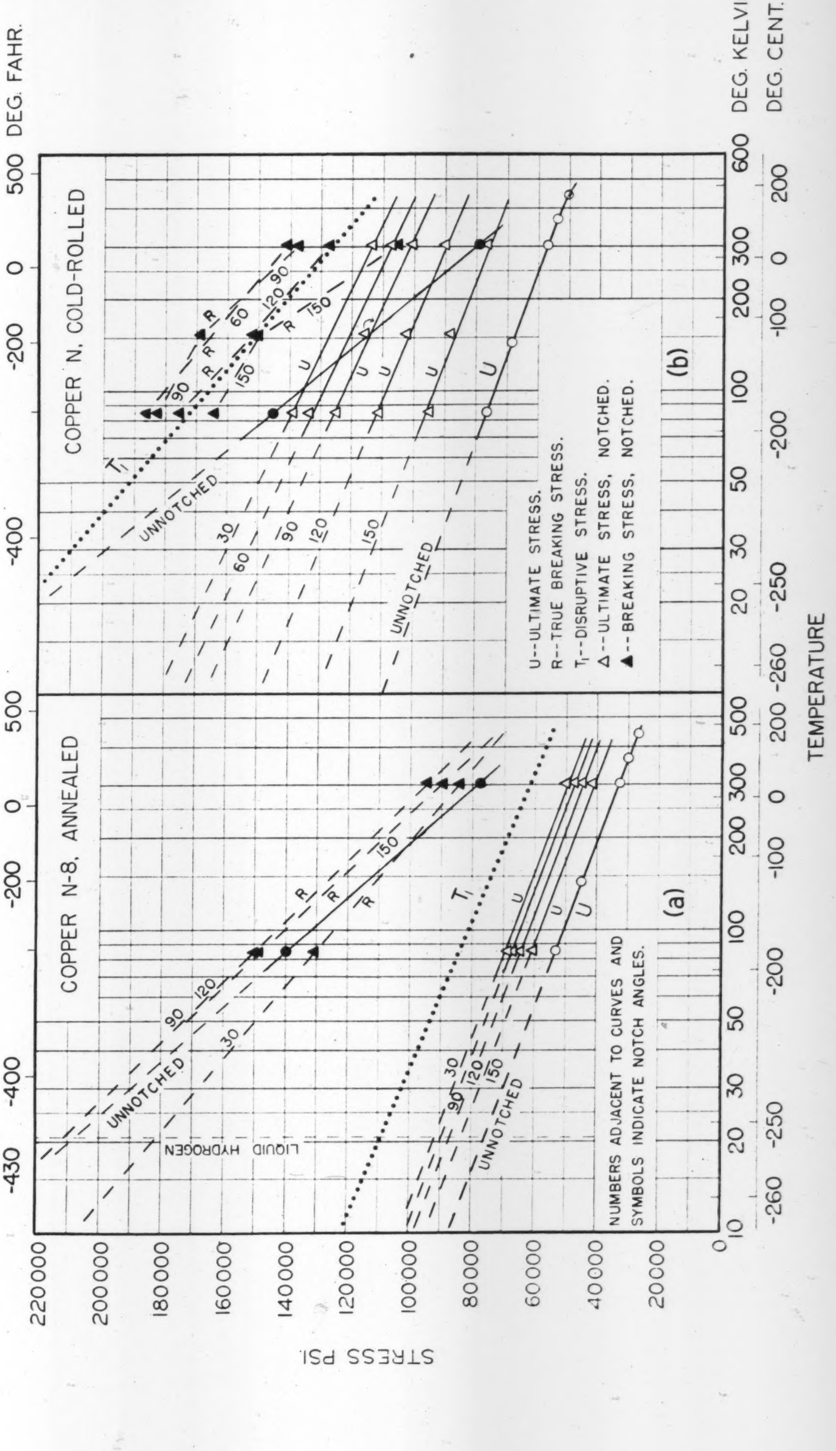

FIG. 20.—Variation of the Strength of Copper with Temperature Represented on a Scale of Equal Thermodynamic Efficiencies.

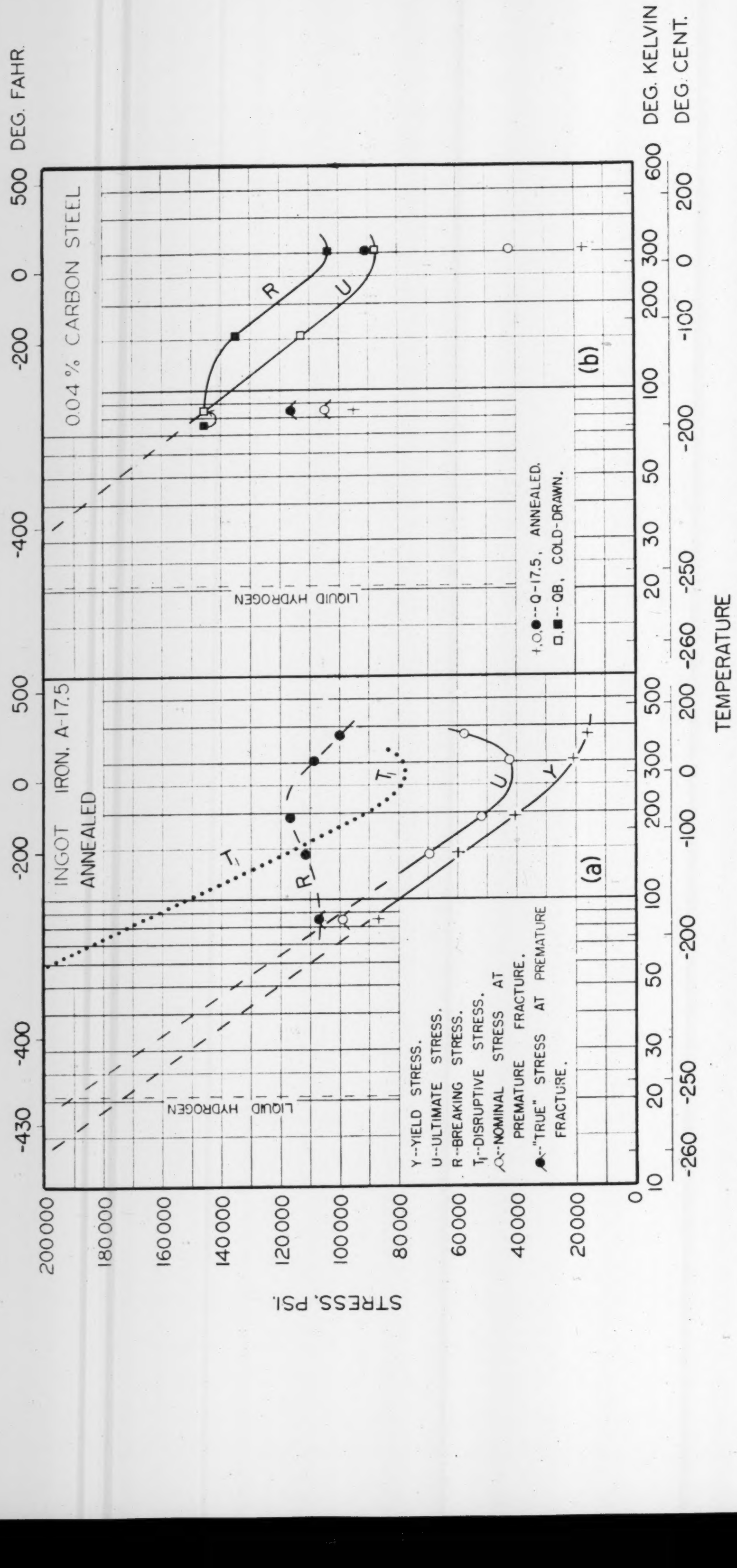

FIG. 21.—Variation of the Strength of Low Carbon Steel with Temperature Represented on a Scale of Equal Thermodynamic Efficiencies.

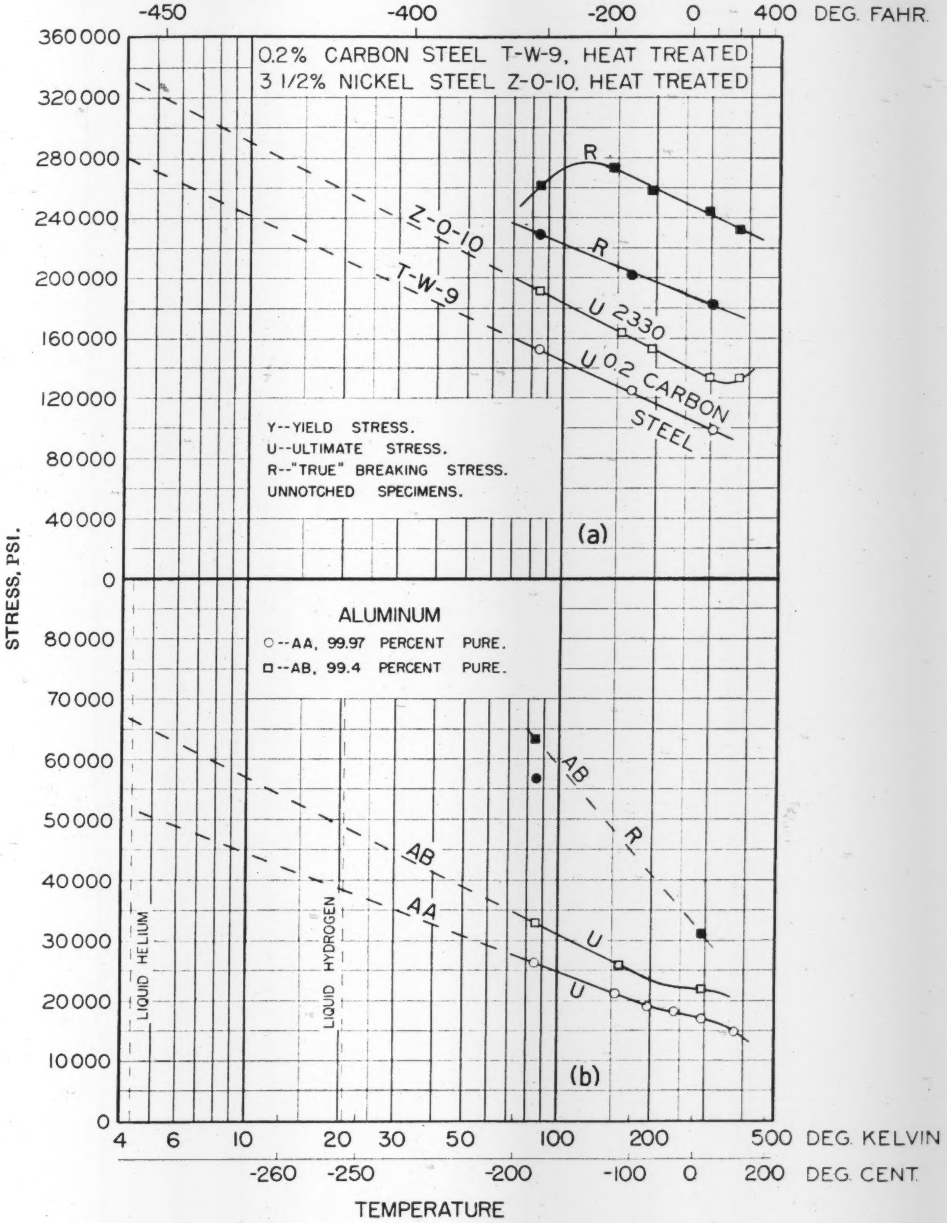

FIG. 22.—Variation of the Strength of Steels and Aluminum with Temperature Represented on a Scale of Equal Thermodynamic Efficiencies.

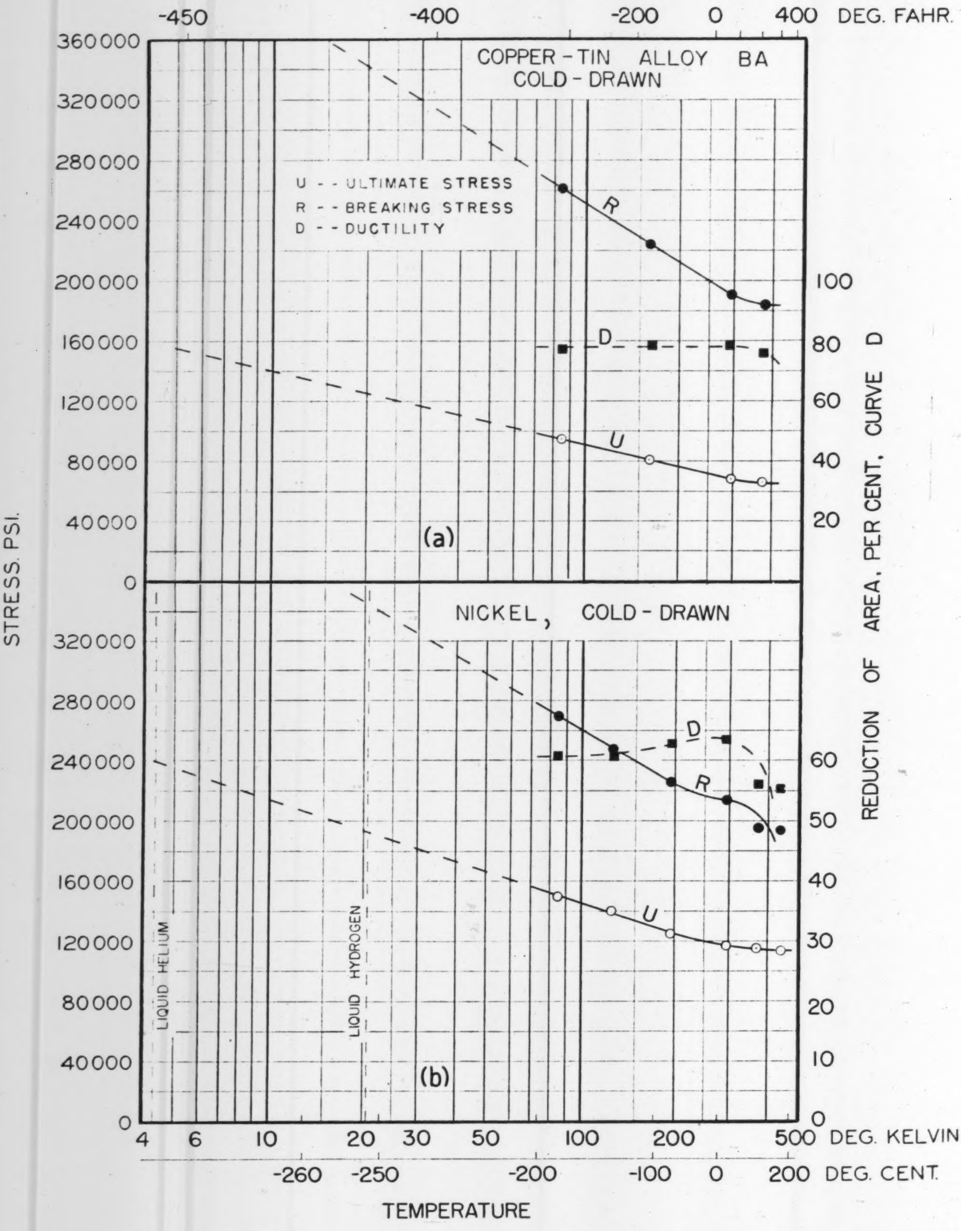

Fig. 23.—Variation of the Strength of Copper-Tin Alloy and Nickel with Temperature Represented on a Scale of Equal Thermodynamic Efficiencies.

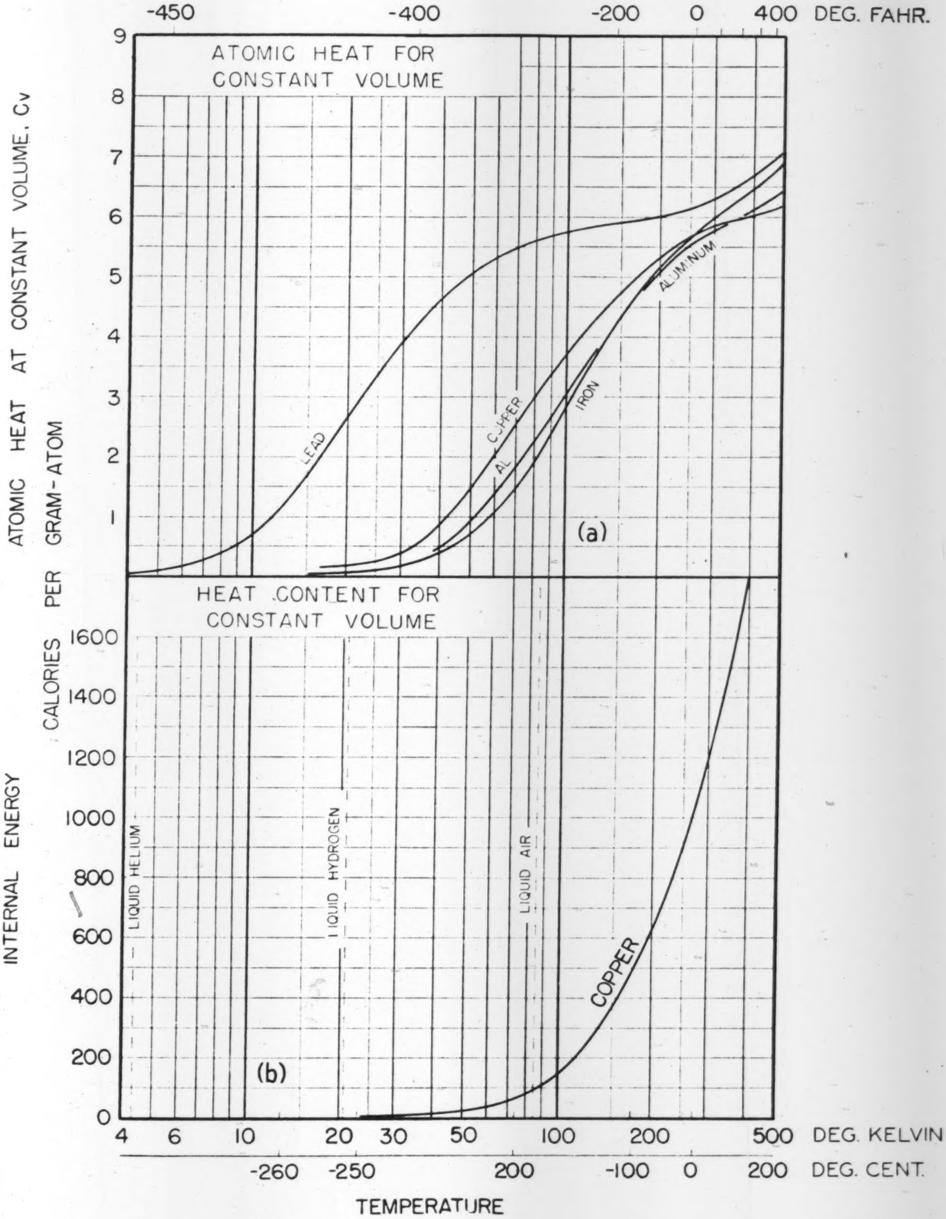

FIG. 24.—Variation of Atomic Heat and Total Heat Content of Some Commercial Metals with Temperature Represented on a Scale of Equal Thermodynamic Efficiencies.

perature on a scale with equal intervals corresponding to equal thermodynamic efficiencies it is not necessary to use the system of numbers proposed by Kelvin. It is necessary merely to plot on a logarithmic scale numbers taken from Kelvin's arithmetical scale. The scale thus obtained, which is in principle Kelvin's original thermodynamic scale, extends from $-\infty$ to $+\infty$.

Consideration of the relationship between Kelvin's two scales in connection with the accelerated rise of the curves of strength of metals in Figs. 17 and 18 led to the idea that the variation of the strength of metals might be represented better on Kelvin's original scale than on his second scale. It appeared possible that an accelerated rise might not be found if strength values were plotted on a temperature scale that extends from $-\infty$ to $+\infty$. This idea has been applied in Figs. 19 to 23. In future references, this scale will be termed a "scale of equal thermodynamic efficiencies."

Diagrams for the same four metals that are represented in Figs. 17 and 18 are shown in Figs. 19, 20, and 21. Attention will be given first to results obtained with unnotched specimens. The yield stress and ultimate stress for either annealed or cold-drawn monel metal (Fig. 19 (*a*)) evidently vary linearly with the temperature (represented on this scale), at least throughout a range from +100 C. down to −188 C. A single straight line, moreover, may be used to represent the variation of the breaking stresses for both annealed and cold-drawn monel metal.

The ultimate stress of annealed copper (Fig. 20) also varies linearly with the temperature, at least from −188 C. up to +165 C. For the cold-rolled copper, the linear relationship extends at least from −188 C. to +100 C.; softening of this severely cold-worked metal evidently begins not far above 100 C. Because the breaking stress of the oxygen-free, high-purity copper can be determined only indirectly (**14**), no conclusions can be drawn at present regarding the variation of the breaking stress with temperature.

Results obtained with the 13 per cent chromium, 2 per cent nickel steel also indicate an (at least) approximately linear variation of the ultimate stress (Fig. 19 (*b*)). Through all the experimental points except those at 123 K. and 145 K. a straight line can be drawn. The points at 123 K. and 145 K. are below the straight line in Fig. 19 (*b*) and below the corresponding curve in Fig. 17 (*b*). It is possible, therefore, that these two points are too low. The breaking stress values for this steel (Fig. 19 (*b*)) are not incompatible with the idea that there is a linear relationship between the breaking stress and temperature (on this scale). The linear relationship for both the ultimate stress and the breaking stress evidently does not continue above room temperature, and the curvature may begin a little below room temperature. The deviation from the linear variation may be attributed to age hardening.

The yield stress of annealed ingot iron (Fig. 21 (*a*)) evidently varies linearly with temperature, at least from −188 C. up to the temperature of solid CO_2 (−78 C.). The ultimate stress may also vary linearly through nearly the same range; at −188 C., however, fracture occurs before the tensile load reaches a true maximum. Not far above the temperature of solid CO_2, the influence of age hardening manifests itself in the curvature of the lines representing both yield stress and ultimate stress. With the cold-drawn 0.04 per cent carbon steel at −188 C. (Fig. 21 (*b*)), the tensile load reached a true maximum just at fracture. Although more experimental points are needed to establish definitely the variation of the ultimate stress with temperature, the evidence is not incompatible with the view that the curve is approximately correct as drawn, and that the ultimate stress varies linearly at least from −188 to −78 C.

As the yield stresses of the cold-drawn monel metal, cold-rolled copper, and cold-drawn ingot iron, nearly coincide with the ultimate stresses, the evidence suggests that the resistance of metals to plastic deformation, as expressed by either the yield stress or the ultimate stress, varies linearly with temperature (represented on a scale of equal thermodynamic efficiencies), provided that this relationship is not obscured by the influence of structural changes such as age-hardening. The exactness of this linear relationship is remarkable. In many tests made after discovery of the relationship, values of the ultimate stress were predicted accurately either by long extrapolation downward to −188 C. or by interpolation between the extremes of the temperature range. For copper, monel metal, and other metals to be considered later, ultimate stress values were frequently predicted within about 0.5 per cent.

With the two steels represented in Fig. 22 (*a*), an exactly linear relationship was found, at least between −188 C. and room temperature. The nickel steel is the same that was used by Rosenberg (**20**), and the experimental points at −78 C. and at room temperature are derived from his data. The other points are results obtained by the authors. By extrapolation from Rosenberg's results, the ultimate stress at −188 C. was predicted within 0.5 per cent. By interpolation, the ultimate stress at −118 C. was then predicted with equal accuracy. The 0.2 per cent carbon steel is the same that was used by the authors in the investigation described in a previous paper (**14**). From the values of ultimate stress at room temperature and −188 C., the value at −105 C. was predicted accurately.

An exactly linear relationship was also found with cold-drawn copper-tin alloy (Fig. 23 (*a*)) for both ultimate stress and breaking stress, at least between −188 C. and room temperature. Above room temperature, a curved relationship begins, probably because of age hardening. With cold-drawn nickel (Fig. 23 (*b*)) the variation of both ultimate stress and breaking stress is linear, at least between −188 C. and −78 C. Not far above that range, the effects of age hardening appear. With cold-drawn aluminum also (Fig. 22 (*b*)) the ultimate stress varies linearly, at least between −188 C. and −78 C. The curvilinear relationship beginning above this range probably is due to age hardening. Above room temperature, however, this severely cold-worked metal begins to soften.

The evidence in Figs. 19 to 23 indicates that when the ultimate stress varies linearly with the temperature, the breaking stress also varies linearly. The variation of the breaking stress is due not only to the influence of temperature on the technical cohesive strength but also to the influence of temperature on three other variables: (*a*) the total plastic deformation (ductility), (*b*) the radial stress induced by the local contraction, and (*c*) the rate of work hardening during the tension test. With monel metal, copper, aluminum, copper-tin alloy, and nickel, the reduction of cross-sectional area due to plastic deformation changes little between room temperature and −188 C. Consequently, the influence of factors (*a*) and (*b*) on the variation of the breaking stress of these metals with temperature is slight, and the variation of the breaking stress is due almost entirely to the influence of temperature and of the variable rate of work hardening on a specific technical cohesion limit. A linear variation of the breaking stress of such a metal thus represents a nearly linear variation of the technical cohesive strength. This linear variation, however, is not a function of temperature alone, but is a function of both temperature and the associated variable, the rate of work hardening. The linear variation of the breaking stress suggests, though it does not necessarily imply, a linear variation of the rate of work hardening and a linear variation of the technical cohesive strength with temperature alone. This suggestion receives support from the evidence obtained with notched specimens.

Results obtained with notched specimens are shown in Figs. 19 and 20. The ultimate stress values obtained with notched specimens of cold-rolled copper (Fig. 20 (*b*)), although they naturally show more scatter than do the values obtained with unnotched specimens, indicate that for each specific notch, the ultimate stress varies linearly with the temperature. The introduction of transverse radial stress, therefore, does not disturb the linear variation of the resistance to plastic deformation with temperature. Because of the wide scatter of the breaking stress values, the true variation of the breaking stress cannot be determined. The evidence, however, is not incompatible with the view that this variation is linear. Support for this view is given by the fact that the variation of the ultimate stress remains linear as the radial-stress ratio is increased by decreasing the notch angle. The linear relationship persists as the line of ultimate stresses is raised and approaches the corresponding line of breaking stresses. If the radial-stress ratio were increased to 1.0, the two lines would coincide, and would then represent the disruptive stress. The evidence in Fig. 20 (*b*) thus suggests that the disruptive stress varies linearly with the temperature. The line T_1, therefore, is drawn straight between points representing values for the disruptive stress at room temperature and at −188 C. (Fig. 1).

The less complete evidence for annealed copper (Fig. 20 (*a*)) and for monel metal (Fig. 19 (*a*)) also suggests a linear variation of the ultimate stress of notched specimens and a linear variation of the technical cohesive strength. Lines T_1, therefore, have been drawn straight between points corresponding to the positions of T_1 in Figs. 1 and 3, respectively.

With notched specimens of 13 per cent chromium, 2 per cent nickel steel (Fig. 19 (*b*)), the ultimate stress corresponding to a constant notch angle evidently varies linearly throughout a range from room temperature down to the temperature at which fracture becomes premature. With a notch angle of 150 deg., the variation evidently is linear at least from room temperature down to −188 C. With notch angles 120 and 90 deg., the linear variation cannot be followed down to −188 C. because fracture at that temperature is "premature." The variation of the ultimate stress evidently is linear, below room temperature, throughout the range of temperature within which the ultimate stress can be determined. The breaking stress for a given notch is approximately linear from room temperature down to the temperature at which fracture becomes premature. As the radial-stress ratio is increased by decreasing the notch angle, the line of ultimate stresses rises and approaches the corresponding line of breaking stresses.

The line (T_1) representing the variation of the disruptive stress has been drawn in Fig. 19 (*b*) through points corresponding to the values of the disruptive stress indicated for four different temperatures in Fig. 5. The evidence thus indicates that the disruptive stress varies (at least approximately) linearly with temperature, throughout a range from room temperature down to −188 C. Curve T_1 for ingot iron (Fig. 21 (*a*)) is similarly based on the values of T_1 obtained from diagrams (*a*), (*b*), and (*c*) of Fig. 7.

The evidence obtained with notched specimens, together with the observed linear variation of the breaking stresses of unnotched specimens, indicates that the technical cohesive strength of metals varies nearly, if not quite, linearly with temperature represented on a scale of equal thermodynamic efficiencies, provided that the linear relationship is not obscured by structural changes. The percentage increase of the technical cohesive strength with decrease of temperature does not differ greatly from the percentage increase of the ultimate stress. For annealed single-phase metals such as copper and monel metal, and for steels, the percentage increase of the technical cohesive strength is less than that of the yield stress. For ingot iron the yield stress is quadrupled by a decrease from room temperature to −188 C., whereas the disruptive stress (T_1) is approximately doubled (Fig. 21). For the other steels, the difference is less.

The evidence in Figs. 19 to 23 gives no hint that the linear increase of strength with decrease of temperature ceases at −188 C. The only evidence in conflict with the idea that the linear relationship continues below this temperature is given in the previously discussed paper by DeHaas and Hadfield (**4**). It seems improbable, however, that all the widely differing metals represented in Figs. 19 to 23 reach an abrupt limit of strength when the temperature is lowered to the liquifying point of the gaseous mixture that constitutes the atmosphere of the earth. The extension of the straight lines in Figs. 19 to 23 below −188 C. is intended merely to indicate the strength values that would be obtained if the linear relationship so continues. It is not intended either to affirm or deny the possibility that the strength may increase without limit throughout the unlimited range of temperature on a scale of equal thermodynamic efficiencies.

The Theoretical Significance of the Linear Variation of the Strength of Metals with Temperature Represented on a Scale of Equal Thermodynamic Efficiencies

Time and space are available for only a brief, inconclusive discussion of the significance of the linear variation of the strength of metals. This linear relationship probably will have considerable influence on the future development of the theory.

A dynamic theory of strength of metals was proposed by Becker (**2**) and elaborated by Orowan (**15, 16, 17, 18, 19**). This theory, however, applies to the rate of creep and to resistance to plastic deformation, not to resistance to fracture. According to Orowan (**19**), variation of the yield stress, on the usual arithmetical temperature scale, is represented by a parabola starting at a finite stress value at "absolute zero." Such a relationship, however, is very different from the linear relationship shown in Figs. 19 to 23. The linear relationship may be expressed by the equation:

$$\begin{aligned} S_A - S &= C\,(\log T - \log T_A) \\ &= C \log T/T_A \end{aligned}$$

where:

S_A represents the strength value at temperature T_A on the Kelvin scale, S represents the strength value at any temperature T, and C is a constant.

This can be placed in the form of an exponential equation. When temperature is represented on a scale with equal intervals corresponding to equal thermodynamic efficiencies, however, strength appears to be linearly related to temperature.

The strength of metals is the only property, known to the authors, that is linearly related to temperature represented in accordance with Kelvin's original thermodynamic scale. Concerning the reason for this linear relationship there is need for intensive study. The idea that both resistance to plastic deformation and resistance to fracture are determined by values of energy rather than by values of force does not appear to account for the linear variation of the yield stress. The linear stress-temperature relationship at present merely suggests two questions, possibly now unanswerable: Does temperature have a theoretically attainable lower limit? Is the scale of equal thermodynamic efficiencies a scale of absolute temperature?

Although Kelvin's arithmetical scale is adequate for the study of the influence of temperature on the strength of metals throughout not too wide a range, his scale of equal thermodynamic efficiencies may be more suitable when the view is extended over a wide range of temperatures, especially over a range of low temperatures. For the wider range, the numbers of Kelvin's arithmetical scale possibly should be viewed in terms of geometric rather than arithmetical progression. Whatever may be the theoretical implication, such a view evidently is of practical value, because of the possibilities of linear extrapolation.

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DISCUSSION

MESSRS. R. G. STURM[1] AND W. L. FINK[1] (*by letter*).—In this paper, which contains so much data on the technical cohesive strength of metals at low temperatures, the authors have made a valuable contribution to the literature. There are, however, a few minor items to which we would like to call attention.

The theoretical stress concentration for elastic stresses is computed on the basis of elastic stress-strain relationships. At the breaking load the strains at and near the point of fracture far exceed those for which the elastic stress-strain relations apply. Consequently for breaking loads, variations in longitudinal strain are expected to cause only slight variations in stress and longitudinal stress concentration factors lose their significance. On the other hand radial stresses are developed in the short reduced section within the notch by virtue of the total reduction in transverse dimensions at this point. Therefore, they would be expected to become more significant for large plastic strains than for small elastic strains. Emphasis may therefore be placed on determining the actual state of triaxial stress at the point of fracture for large amounts of plastic strain.

The curves of Fig. 22(*b*) showing the variation in ultimate strength of aluminum with temperature are drawn with two branches—a linear branch below −78 C. and a curvilinear branch above this temperature. The authors attribute this curvilinear relationship to age-hardening. Inasmuch as the specimens were aluminum of high purity—99.4 per cent and 99.97 per cent—and not age-hardenable aluminum alloys, some explanation other than age hardening would be required to account for a change in the shape of the curve. No age hardening has ever been observed in aluminum of these purities. Moreover, even in the age-hardenable aluminum alloys age hardening does not occur in the neighborhood of −78 C.

This raises the question as to whether the data justify drawing the curves in the manner used by the authors. The deviation from the best straight line through the points would probably be less than the scatter obtained in testing a number of different samples at a given temperature.

MR. A. NADAI.[2]—I was much impressed by the test results reproduced in Figs. 19 and 20 of the paper in which among other quantities the yield, the ultimate, and the breaking stresses were plotted in function of the logarithms of the absolute temperature. In these figures beautiful straight lines expressed this dependence of stress on the logarithms of the absolute temperatures. I am not so familiar with the work done by physicists in connection with the temperature dependence of the flow properties of metals, but if I am correct, they use different scales for representing some of these functions. For example, it has been found by engineers that the function representing the variation of the yield stress at a given strain or of the true stress at the maximum load in a tensile stress-strain curve (at a given temperature) can be represented by the logarithmic function

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$$\sigma = \sigma_0 \log_e \frac{v}{v_0} \quad \ldots\ldots\ldots(a)$$

where σ designates the true stress, v the plastic rate of flow, and σ_0 and v_0 are material constants. It has furthermore been claimed by physicists[3] that the important material constant v_0 is proportional to a factor $e^{\frac{Q}{RT}}$. Therefore, if the logarithms of the intercepts v_0 in the log rate of flow v *versus* stress σ diagram (our Eq. *a*) are plotted in function of the reciprocal absolute temperatures, it was claimed that the theory predicts a straight line. Perhaps the authors would be good enough to explain to us whether other physical reasons were speaking in favor of using log T instead of $1/T$ in similar linearizing plots.

I should like to comment on another question referring to the possibility of producing in a bar with a notch three equal stresses in tension or a distribution which would closely enough approach such a state of stress. I am sorry to say that I am not prepared for a discussion, and I have not looked up the former papers of the authors devoted to the same subject, but I remember from Kunze's papers that he used round specimens with very deeply cut circumferential notches in which very little material was left in the constricted portion of the test bar. Several years ago MacGregor and the writer came across this type of test specimen when we made a few photoelastic tests with deeply notched specimens in tension. We were not able to investigate the technical cohesive strength, but the photoelastic tests showed us the stress distribution in the neck, if I remember correctly. From these tests it was not easy to see how one can produce or how one can approach, even in the elastic state in the minimum cross-section of a notched bar, a stress distribution which would consist near the axis of the bar of hydrostatic tension with three equal principal stresses when at the same time a finite radius of curvature is maintained at the bottom of the constriction and the width or the diameter of the minimum cross-section tends to approach the limiting value zero.

MESSRS. D. J. MCADAM[4] and R. W. MEBS[4] (*authors' closure, by letter*).—The authors agree with Messrs. Sturm and Fink that, at fracture of a notched specimen of sufficient ductility, the original concentration of longitudinal stress is largely obliterated, and that in a properly designed specimen the actual longitudinal stress at fracture probably differs little from the mean stress. Whether the radial stress increases with the plastic deformation, however, depends on the change in the notch depth, notch angle, and notch radius. This has been discussed by the authors in another paper (**14**).[5] The authors also agree that it would be desirable to know quantitatively the stress distribution at fracture. However, the conclusion of the authors that the technical cohesion limit tends to increase with increase in the radial stress ratio in the manner indicated by the diagrams, is not affected by the absence of this quantitative knowledge. A careful study of the evidence, with comparison of the *mean* breaking stresses of notched and unnotched specimens, can lead to no other conclusion, unless one wishes to assert that the actual stress at fracture of a notched specimen may be far below the *mean* stress at fracture, and even far below the mean stress at yield strength (**11, 12, 14**).

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[3] W. Kauzmann, "Flow of Solid Metals from the Standpoint of the Chemical-Rate Theory," Am. Inst. Mining and Metallurgical Engrs., 1943 Preprint. In this paper reference is made to investigations by R. Becker, H. Eyring, E. Orowan and others on the temperature dependence. Compare particularly Fig. 8 in the paper by Kauzmann.

[4] Metallurgist, and Associate Metallurgist, respectively, National Bureau of Standards, Washington, D. C.

[5] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to the paper, see p. 702.

With regard to the curves for the strength of aluminum (Fig. 22*b*), some of the experimental points represent averages of two closely agreeing values. The course of the curves, therefore, is as indicated. The fact that pure aluminum is not age-hardenable by heat treatment does not preclude the possibility that it may be subject to strain aging. Mr. Richard Miller of the United States Steel Corp. informed the writer that he had observed definite strain aging in a single crystal of high purity aluminum. Iron of even greater purity than the aluminum shows definite strain aging, beginning at temperatures considerably below 0 C. Copper of equal purity with the aluminum gives straight lines, as shown in Fig. 20. It seems logical to attribute the different behavior of aluminum to the structural changes that have been called strain aging.

With regard to Mr. Nadai's comments, the reason for using a temperature scale based on Kelvin's original thermodynamic scale is given in the paper. The paper also points out that the linear relationship found is not compatible with an equation based on the Becker-Orowan theory. The relationship could not be linear both for log T and $1/T$. The authors have shown a linear relashipship between strength and log T.

Mr. Nadai's comment on the possibility of producing polarsymmetric tension by loading a notched bar would seem to indicate that he has not read any of the papers on technical cohesion either by Kuntze or by the authors. Notched bars are used as a means of obtaining transverse radial tensile stress. Different methods of extrapolation have been used to obtain values for breaking stress under polarsymmetric tension. The conclusions of the authors however, do not depend on the validity of any method of extrapolation. The evidence proves that the technical cohesion limit tends to increase with increase in the radial stress ratio (**11, 12, 14**).

INTERPRETATION OF CREEP TEST DATA

By P. G. McVetty[1]

Synopsis

The urgent need for creep data on a wide variety of heat-resisting alloys has emphasized the importance of the problem of producing the maximum useful output from available creep-testing equipment. The basic objectives of all high-temperature testing are the comparison of materials and the choice of safe working stresses. Creep testing occupies an important place in this field since it provides data for a large number of applications in which the criterion of failure is not actual rupture but the attainment of a specified small allowable deformation within the life of the material in service.

This paper bases the interpretation of creep test data upon the establishment of relations among the four variables—temperature, stress, deformation, and time. Various factors affecting these relations are considered, particularly the effects of stress and temperature upon the deformation-time (creep) curve. Methods are suggested for determining the necessary duration of a test and for extrapolation from short to long times and from high to low stresses. Simple, practical interpolation and extrapolation methods are developed for the presentation of creep results in the form of tabulated data or charts covering any desired ranges of stress and temperature and using relatively few creep tests. These methods are adapted particularly to the correlation of data from various sources and the correction of preliminary estimates when more data become available.

To illustrate the proposed method, creep curves from eight tests of a 35 per cent nickel, 19 per cent chromium heat-resisting alloy at three temperatures are given. From these are developed sample charts for guidance in the choice of working stresses and the comparison of materials. Since the purpose of the paper is to describe and illustrate a method of analysis rather than to give specific data on any material, the detailed information on chemical analysis, heat treatment, etc., usually required has been omitted.

During the past twenty years much time and effort have been expended in the conduct of tests involving the effects of long exposure to stress at elevated temperatures upon metals and alloys. In general these tests attempt to simulate service conditions and to establish relations among the four variables—temperature, stress, deformation, and time. Variations in service conditions suggest different types of tests which might be used to establish these relations. In choosing one of them for this discussion, there is no intention to imply that other tests and other methods of interpretation cannot be used to reach the desired objectives. The term

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"creep" has been applied to the gradual deformation or flow of materials in the direction of an applied stress, usually over a relatively long period of time, and at elevated temperatures. To secure the most effective utilization of creep test data, it is important that methods of interpretation be reviewed occasionally to follow changing concepts of high-temperature problems. Also, appraisals of existing alloys must be revised periodically to ensure proper consideration of the ever-increasing supply of creep test data and fair comparison of old and new alloys.

The Tension Creep Test

The first creep tests made to obtain specific design information were conducted by Dickenson and the results published in 1922 (**1**).[2] He used very simple apparatus in which stress and temperature were maintained approximately constant while deformation was measured over a long period of time. Details of equipment (**2**) and testing technique[3] have been improved in many ways, but the basic principles of Dickenson's tension creep test remain unchanged. It is more widely used today than any other tests as the basis for reliable estimates of deformation after long periods of time at high temperatures.

Any investigation of time effects raises the question of relative merits of accelerated tests and those in which the duration of the test approaches that of the expected service life. The difficulty and cost, both in time and money, of long-time creep tests have stimulated efforts to obtain similar information in a shorter time. Considerable progress has been made in the development of rapid tests for comparison of materials and, to some extent, for choice of working stresses. An early attempt to provide a basis for choice of working stresses is due to Barr and Bardgett (**3**). Particular reference is made to the discussions of A. M. Roberts (**3**, p. 356) and H. J. Tapsell (**3**, p. 361) which show why tests limited to a duration of two days cannot be satisfactory for the purpose intended. A realization of the value of the test when extended to longer time periods is shown in later studies (**4**). This type of test is adapted to high-temperature bolting problems in which the length remains practically constant while part of the original elastic stretch of the bolt is converted into plastic deformation with a corresponding stress reduction. This change in stress affects the continued tightness of flanged joints operating at high temperatures.

A somewhat similar method of acceleration was proposed by Rohn (**5**). In this method, the stress is unchanged while the length is maintained constant by reducing the temperature, and creep is balanced by thermal contraction. This method was also tried out in this country (**6**), but it has not succeeded in establishing a satisfactory basis for estimating creep behavior over long periods of time. Other tests such as high-temperature stress-rupture, fatigue, impact, torsion (shear), etc., are made in simulation of specific service conditions, but they cannot be considered here. It is necessary to limit this discussion to applications in which stress and temperature are nearly constant. A certain amount of variation is common in practice, but it is customary to assume continuous exposure to the maximum stress and temperature. Departures from this assumption in service add to the factor of safety. As our knowledge of creep behavior increases, it may be possible to make allowance for stress and temperature fluctuations to increase stress estimates, but this is hardly justified at the present time.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 726.

[3] Standard Recommended Practice for Conducting Long-Time High-Temperature Tension Tests of Metallic Materials (E 22 – 41), 1942 Book of A.S.T.M. Standards, Part I, p. 930.

Under these simplified conditions, we may consider two important problems:

1. For a given allowable deformation within some specified service life, what is the maximum stress that can be used?

2. On the basis of creep strength at a given temperature, what are the relative merits of available alloys?

It is the purpose of this paper to show:

(*1*) That these simple questions can be answered by suitable interpretation of creep test data,

(*2*) that reasonable estimates may be made from very few tests,

(*3*) that the reliability of estimates may be improved by a few additional tests on the more promising materials, and

(*4*) That the same form of analysis is adapted to the preparation of charts for aid in the choice of working stresses and for the comparison and selection of materials.

Factors Affecting the Conduct of Creep Tests and Interpretation of Creep Test Data

One of the difficulties that has delayed final analysis of creep test data is the large number of variables which affect the manufacture and treatment of alloys, the tests used to evaluate them, and the estimates of conditions under which they will be used. While it may be desirable, theoretically, to extend the duration of tests to cover an appreciable portion of the expected service life, new alloys are being developed too rapidly to permit postponement of their use to await completion of such tests. It is necessary to limit the duration of tests for practical reasons, and methods will be discussed later for setting these limits. Granting that extrapolation with respect to time introduces innumerable objections, it may be considered a necessary evil and treated accordingly.

It is beyond the scope of this paper to discuss in detail the many factors which affect conclusions drawn from creep tests. If one may prescribe an absolute minimum in required reading before consideration of the interpretation problem, the 1939 Howe Memorial Lecture (7) is suggested to stimulate the proper viewpoint. Little objection can be raised to Gillett's rather pessimistic review of the difficulties involved. In the present state of our knowledge, any suggested methods of interpretation must be considered tentative. While the inherent difficulties do present a challenge, the ever-increasing number of successful designs for high-temperature service is proof that we need not wait for more and more test data before we begin to use what we have. Even with the admittedly incomplete and inexact analyses of early test data, high-temperature applications have increased rapidly. Failures due to improper choice of materials or overestimates of safe working stresses have been few in number. In many cases, working stresses have been unnecessarily conservative as a result of uncertainty as to the uniformity of materials and the reliability of estimates based on test data. Further progress will be accelerated if a reasonably sound basis for interpretation can be agreed upon.

After careful study of the metallurgical aspects of the problem, Gillett has outlined certain prerequisites which should be observed in preparing material for high-temperature service. It may be impossible to incorporate all of these requirements into purchase specifications, but we may expect the fullest cooperation from the producers of alloy steels when the requirements are known and understood. If now, we adopt the best testing technique available, the problem still remaining may be stated simply: How can the test results be analyzed and put into the most useful form to guide the selection of materials and the choice of safe working stresses? Solution of this problem requires a philosophy which rates extrapolation as a necessary evil, improves conditions which can be improved, and attempts to make the best of the unfavorable conditions which remain. Unless design is to remain stagnant, some assumptions are required. If experience shows that revisions are necessary from time to time, any form of tentative analysis can be improved to keep it in agreement with the best available information. In this way, constructive criticism improves or replaces each element of the whole analysis as it is found to have outlived its usefulness.

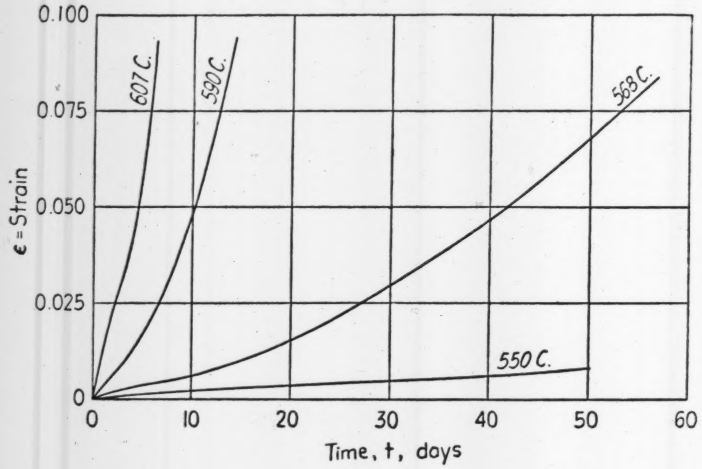

Fig. 1.—Creep Curves for Forged 0.39 per cent Carbon Steel, Stress—3 tons per sq. in. (H. J. Tapsell and A. E. Johnson).

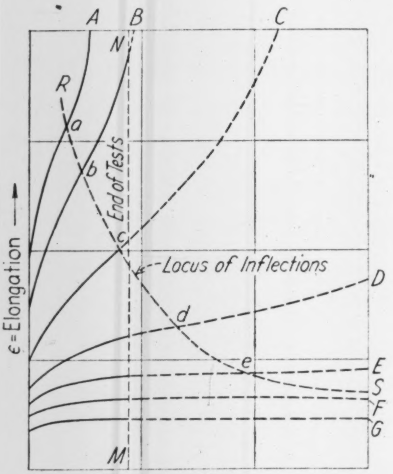

Fig. 2.—Extrapolated Creep Curves.

The importance of the application of creep test data to practical problems has been recognized and many papers have been written on this subject. Reference to a few of them will indicate some of the various points of view from which the problem has been studied (8-22).

An attempt to correlate this material would do much to clear up uncertainties introduced by divergent viewpoints. It may be more satisfactory, however, to outline, as simply as possible, practical methods which have been developed and used to convert creep test data into charts convenient for subsequent use.

TYPES OF CREEP CURVES

Leaving abnormal varieties of creep curves for later discussion, it is necessary to consider at least two types to express the results of creep tests at constant stress and temperature. These are illustrated in Figs. 1 and 2. In covering the desired ranges of stress and temperature variation, it is possible to conduct tests at several temperatures at the same stress or several stresses at the same temperature. For various reasons, the author prefers the latter, but either method may be used. Figure 1 (23) represents a family of creep curves for various temperatures at the same stress. Under suitable conditions, a family of curves of similar appearance could represent various stresses at the same temperature. A striking feature illustrated by Fig. 1 is the major effect on the creep curve of relatively small differences in temperature. The increase from 550 C. to 568 C. at the same stress changes the character of the curve from one which might have a fairly long life to one giving every evidence of early failure. Tests at 1000 F. (540 C.) have shown that a temperature rise of as little as 6 F. can double the creep rate. From data of this kind, it is not difficult to prove the importance of close temperature control during creep tests and the unreliability of tests in which this precaution is not observed.

Examination of Fig. 1 shows an unstable condition, with creep rate increasing, cross-section diminishing, stress for the same load increasing and an expected aggravation of these unfavorable conditions when necking starts. The use of materials under these conditions must be restricted to applications involving relatively short service life and large allowable deformation. Predictions of deformation in service from such curves are necessarily unreliable.

The conditions represented in Fig. 2 show a transition from the type of curve illustrated in Fig. 1 to one in which the alloy may be expected to approach dimensional stability during all, or a large part of its life in service. Predictions of deformation within the expected service life increase in reliability as the form of creep curve progresses from *A* to *G*. In appearance, Figs. 1 and 2 are quite different; actually, the essential difference is only one of emphasis upon the first or third stages of creep. Both types of curves (Fig. 1 and Fig. 2) might be obtained from tests of the same material by extending the tests over sufficient stress and temperature ranges. The simplest explanation may be based on Bailey's definition of creep (24) as the net effect of the two opposing forces of strain-hardening and annealing. In Fig. 1, the annealing effect predominates, while in Fig. 2 the influence of the two forces gradually changes from predominance of annealing in curve A to predominance of strain-hardening in curve G. Available evidence indicates that most creep curves, if sufficiently prolonged, would have the form shown in *D* of Fig. 2 (25). During the first stage of creep, the rate of deformation is decreasing rapidly due to strain hardening. In the third stage, the rate of deformation increases as the time of exposure to the high temperature is increased, with a resulting cumulative annealing effect. In the second stage, the two opposing forces of strain-hardening and annealing are approximately balanced and a period of time at a nearly constant rate results. The importance of the approximately steady state existing in the second stage will be discussed later.

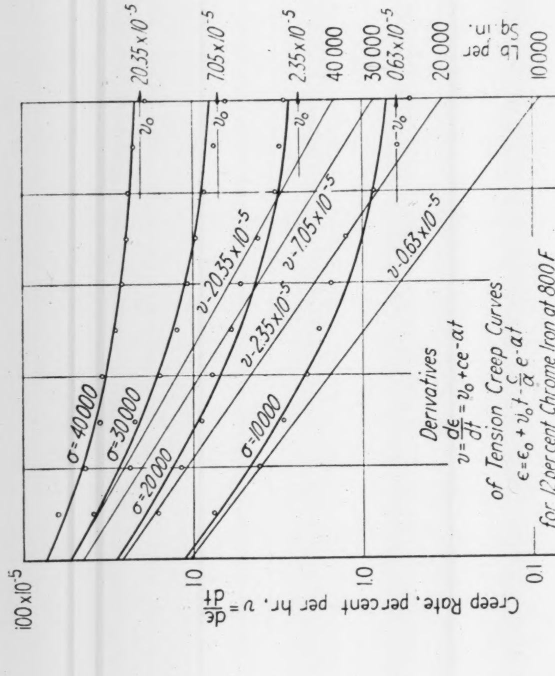

FIG. 3.—Derivatives of Tension Creep Curves. (Based on Data from J. J. Kanter, Crane Co.)

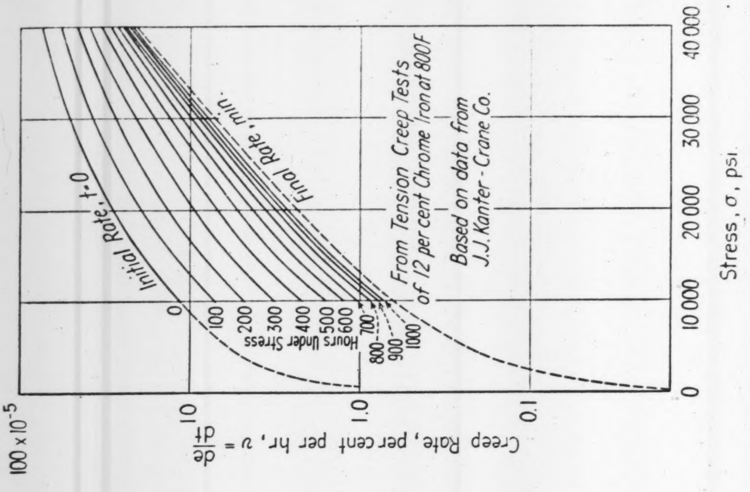

FIG. 4.—Stress - Creep Rate - Time Relations.

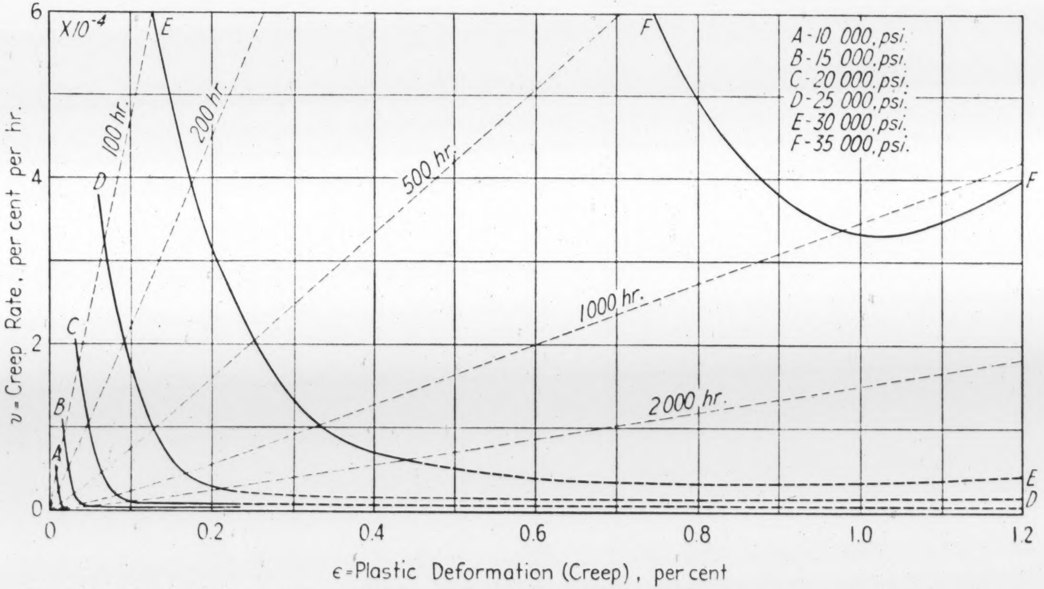

Fig. 5.—Relations Between Creep and Creep-Rate (Method of A. Nadai). From tension creep tests of stainless iron at 850 F. (455 C.).

ANALYSIS OF CREEP TEST DATA

In most cases, the duration of creep tests is restricted to a time which is short in relation to the expected life of the material in service. This is shown in Fig. 2 by the line *MN* which separarates that part of our knowledge of creep curves backed by actual experiments from the part deduced from the study of all available information. It was predicted by the author in 1933 (**25**) that curves of the form represented by *G* in Fig. 2 would pass through an inflection point if sufficiently prolonged. The line *R-S* was suggested as the probable locus of such inflection points at various stresses. The recently published (**26**) creep curves representing tests prolonged to 100,000 hr. tend to verify these predictions. If it were possible to express accurately the deformation-time relation indicated by the line *R-S*, the reliability of extrapolation of creep data would be improved. Two methods have been proposed to indicate the minimum creep rate at the inflection point and the rate of change before and after this minimum. In 1933, the author suggested (**12**, Figs. 10, 12) two charts reproduced in Figs. 3 and 4. Figure 3 shows a method of estimating the minimum creep rate by plotting rates as functions of time at various stresses at the same temperature. In many cases, a constant rate is reached after a relatively short time and little is gained by prolonging the test. Figure 4 expresses the time-stress-creep rate relations in another way. The difference between the curve representing creep rates at the end of any time during the tests and estimated final minimum creep rates indicates whether or not the test should be prolonged.

Another valuable guide was suggested by Nadai in 1938 (**22**, Fig. 13). Figure 5 represents this suggestion as a plot of creep rates as functions of deformations. Since the creep rate v_0 at any instant may be represented as the derivative of the elongation, ϵ, with respect to the time, t, these curves show the relation between $\frac{d\epsilon}{dt}$ and ϵ. This type of chart is particularly valuable for study of the probable effects of extrapolation of creep curves.

It will be noted that the upper curve shows the creep rate decreasing to a minimum value and then increasing. This illustrates the variation in the creep rate of curve A in Fig. 2 as the curve passes through the inflection point. As the stress decreases, that part of the deformation, ϵ, produced at a nearly constant rate, v, increases. Since this deformation proceeds at decreasing rates as the stress is reduced, each succeeding curve shows greatly increasing time at nearly constant creep rates. Since service stresses are usually lower than test stresses, and since it is doubtful if low-stress curves will cross those for higher stresses, it is apparent that very long times at nearly constant creep rates may be expected at the low stresses used in service. This helps to justify what may appear to be excessive extrapolation of low-stress creep curves with respect to time. It tends also to alleviate the fear that a sudden increase in creep rate leading to early failure may be expected at any time.

It has been shown previously (**12**) that the creep curve may be replaced by its tangent or asymptote for extrapolation at low stresses. The reliability of this assumption depends upon the attention paid to the contributing factors which have been discussed. Until a better expression is developed, we may use for tests at constant temperature a tentative relation previously developed (**12**) to connect the deformation with the time:

$$\epsilon = \epsilon_0 + v_0 t - \frac{c}{\alpha} e^{-\alpha t} \quad \ldots . (1)$$

where:

ϵ = plastic deformation (creep),
$v = \frac{d\epsilon}{dt}$ = creep rate,
v_0 = minimum creep rate,
t = time,
e = base of Napierian logarithms, and
c and α = constants depending upon the material and the temperature.

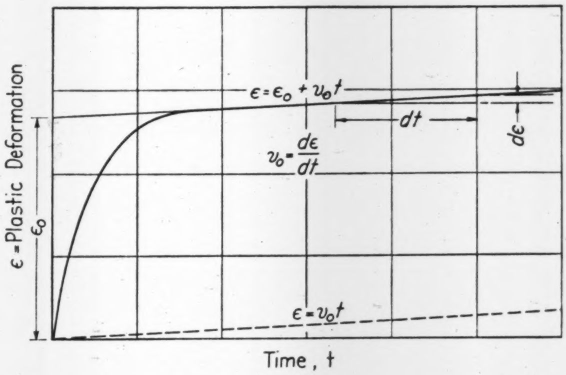

FIG. 6.—The Tension Creep Curve. Plastic deformation (creep) as a function of time.

In this expression, the first two terms define the asymptote while the exponential term serves to define approximately the relations existing in the first stage of creep. Since we are now concerned primarily with extrapolation to times representing the expected service life, it is necessary only to use the asymptote equation:

$$\epsilon = \epsilon_0 + v_0 t \quad \ldots\ldots (2)$$

This curve is shown in Fig. 6 in comparison with the dotted curve representing neglect of the intercept, ϵ_0, in some methods of interpretation.

For all practical purposes, the final tangent of a test of reasonable duration may often be considered the same as the asymptote. The effects of stress and temperature upon extrapolated creep curves may then be studied by considering their effects upon the two constants, ϵ_0 and v_0, which locate the extrapolated curve. The familiar use of the creep rate, v_0, to express completely the creep properties of a material at a given stress and temperature, is permissible only if the intercept ϵ_0 is small compared to the total deformation, ϵ, at the end of the service life, t. When the creep rate is relatively large, as it is in many creep tests, the error resulting from disregard of the intercept, ϵ_0, is not serious. It means that extrapolation is assumed to follow the dotted curve, $\epsilon = v_0 t$, in Fig. 6, instead of the actual tangent or asymptote $\epsilon = \epsilon_0 + v_0 t$. It is obvious that the effect of this simplification depends upon the relative magnitude of ϵ_0 and $v_0 t$ at the end of the service life. Study of actual creep curves shows that this relation varies considerably under different conditions. One conclusion from such study has an important significance. As the stress or temperature decreases, the ratio of the $v_0 t$ product to the intercept ϵ_0 also decreases. This shows that the importance of the intercept ϵ_0 increases with decreasing stress. When creep rates are estimated at stresses lower than actual test stresses as a part of an estimate of total deformation at working stresses, it may be expected that the intercept ϵ_0 will make up an appreciable part of the total deformation.

The extrapolation of creep curves from the end of the test to a time representing the expected service life may be illustrated in Fig. 7. The horizontal line *AB* represents the maximum allowable deformation which must not be exceeded before the creep curve passes the vertical line *CD*. As the stress is decreased, the extrapolated creep curve intersects the line *AB* at the end of increasing periods of time. If the creep curve falls below *B*, the stress is unnecessarily low. The best choice of stress is the one giving the creep curve passing through *B*.

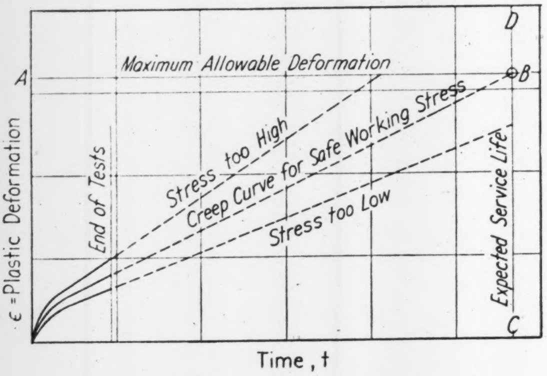

FIG. 7.—Effect of Stress at Constant Temperature upon Extrapolated Tension Creep Curves.

One of the earliest methods of interpretation, which is still used, assumes that a parabolic type of power function expresses the relation between stress and creep rate at constant temperature. This is a very convenient relation because it requires a simple plot of logarithm of stress *versus* logarithm of creep rate and a straight line drawn through the plotted points. The reliability of extrapolation of this curve to low stresses was questioned by Norton (**27**) in 1929, but the convenience of this method has led to considerable use for correlation of creep data (**28**) and, to some extent, for design calculations.

Much evidence has been collected to check the reliability of this procedure at low stresses and a recent paper (**29**) shows quantitatively the errors in stress estimates which may be expected from its use. This paper concludes that the hyperbolic sine relation between stress and creep rate proposed by Nadai (**22**) is the best functional relation now available for estimates of creep rates at low stresses.

As a result of the tendency to disregard the intercept ϵ_0 in the analysis of creep data, our knowledge of the effects of stress and temperature upon this variable is limited. It was shown by the author in 1933 (**12**, Figs. 11, 19, 20) that it could be considered without serious complication. At that time, the relation between ϵ_0 and stress or ϵ_0 and temperature appeared to be approximately exponential. Further study has indicated a relation of the hyperbolic

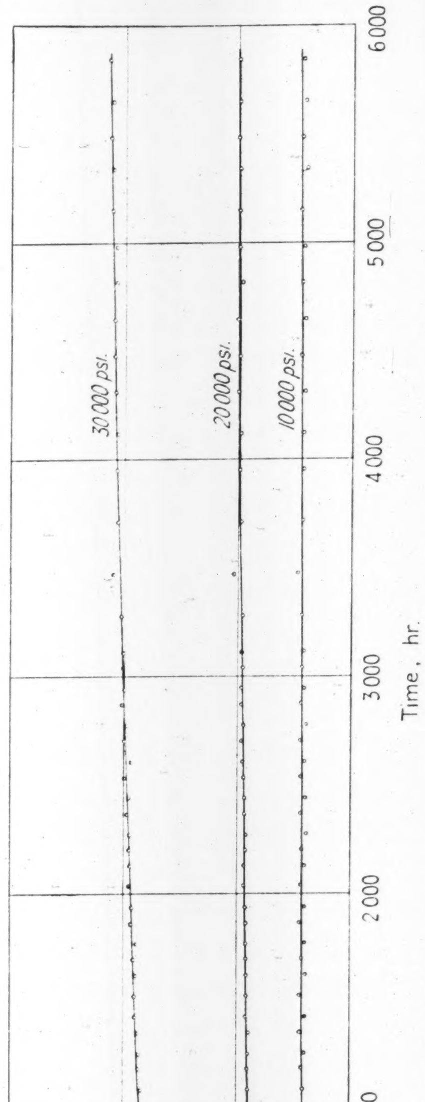

FIG. 8.—Tension Creep Curves for 35 per cent Nickel, 19 per cent Chromium Alloy at 838 F. (448 C.).

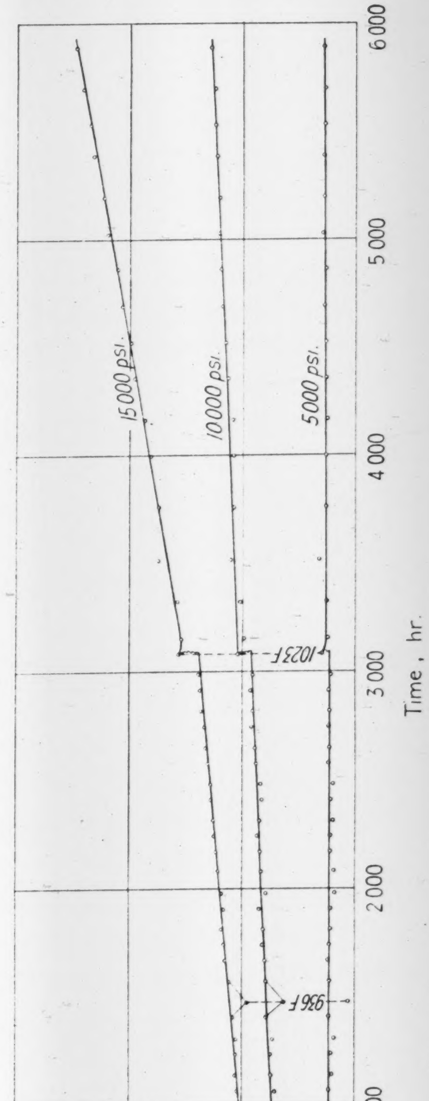

FIG. 9.—Tension Creep Curves for 35 per cent Nickel, 19 per cent Chromium Alloy at 998 F. (537 C.).

sine type at relatively low stresses and temperatures. One element of the problem not previously discussed is the conclusion that ϵ_0 appeares to follow an approximate hyperbolic sine relation in the low stress or temperature field up to a maximum value beyond which it decreases. This possibility is seen easily from inspection of the curves representing the higher stresses and temperatures in Figs. 1 and 2. If for no other reason, this factor alone makes it essential that some tests be conducted in the low stress field to chart the ϵ_0 variation in the region reasonably close to that in which working stresses are located.

From the discussion of the fundamental principles involved, it appears that the objectives of this paper may be realized by conducting tests that will establish the values of minimum creep rates and intercepts over the stress and temperature ranges for which design information or comparative strength data are required.

ILLUSTRATIVE EXAMPLE

To illustrate the principles involved in the proposed method of interpretation the following data are taken from recent tests of a 35 per cent nickel, 19 per cent chromium heat-resisting alloy. Figures 8, 9, and 10 show the creep curves at three temperatures. The tests were continued long enough in each case to permit a reasonably accurate estimate of the position and slope of each curve. The indicated disturbances due to temperature control failures may be considered characteristic of such tests. The observed data, without any corrections, were plotted on large sheets from which the slopes of the final tangents were measured. The effects of departures from constant temperature were then studied. Over-heating usually requires that the test be discarded. If the temperature rise is not excessive, as at 3100 hr. at 998 F. (537 C.) (Fig. 9), it may be desirable to continue the test long enough to measure the subsequent creep rate. If the change in rate is negligible, the vertical shift of the curve may be subtracted from the intercept ϵ_0. The accuracy of the final analysis will depend upon the judgment used in measuring the creep rates and intercepts from the original creep curves which are used to represent each combination of stress and temperature.

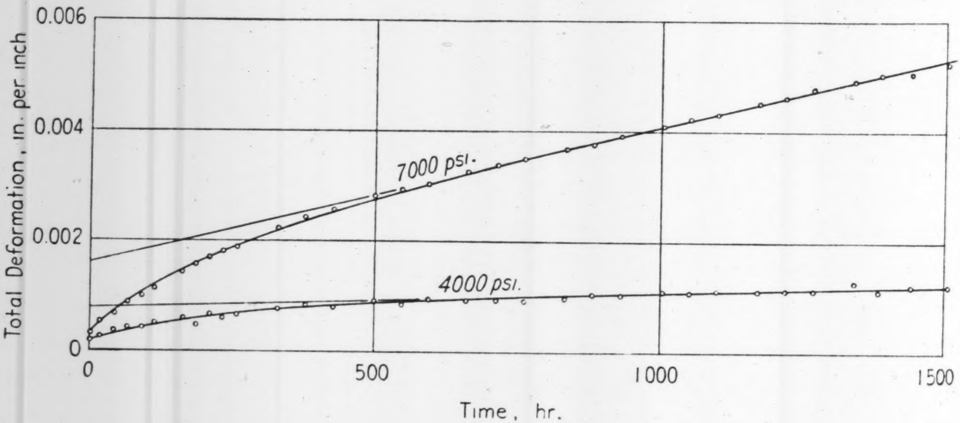

FIG. 10.—Tension Creep Curves for 35 per cent Nickel, 19 per cent Chromium Alloy at 1199 F. (648 C.).

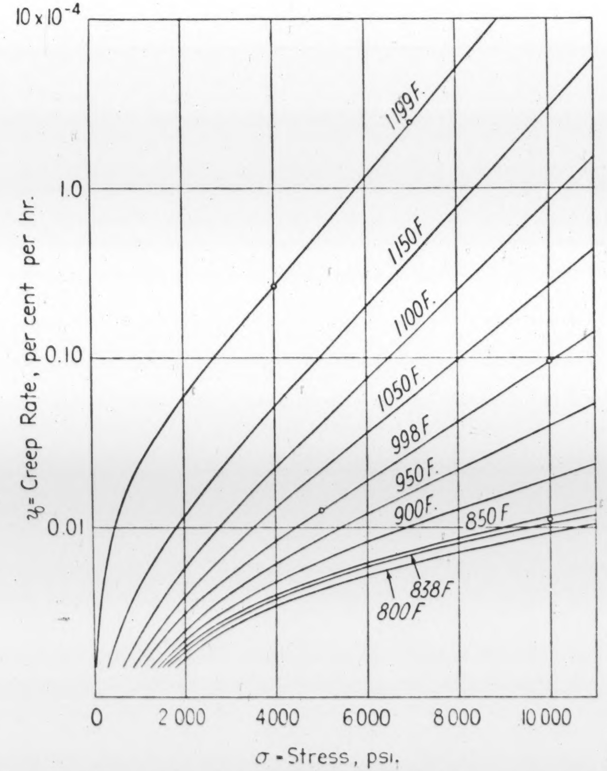

FIG. 11.—Stress-Creep Rate-Temperature Relations. From tension creep tests of 35 per cent nickel, 19 per cent chromium alloy.

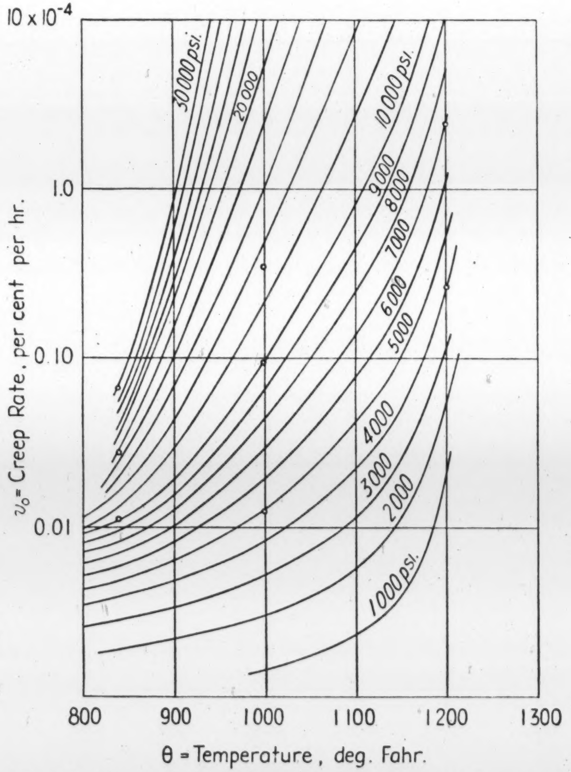

FIG. 12.—Temperature-Creep Rate-Stress Relations. From tension creep tests of 35 per cent nickel, 19 per cent chromium alloy.

The following values were chosen after study of the original curves from which Figs. 8, 9, and 10 were reproduced:

Temperature	Stress, σ, psi.	Creep rate, v_0, per cent per hr., $\times 10^5$	Intercept, ϵ_0, per cent —
838 F. (448 C.)	10 000	0.112	0.0425
	20 000	0.279	0.0919
	30 000	0.668	0.1869
998 F. (537 C.)	5 000	0.127	0.0185
	10 000	0.955	0.0607
	15 000	3.497	0.0281
1199 F. (648 C.)	4 000	2.67	0.08
	7 000	24.5	0.16

It has been shown in a recent paper (**29**) that the stress-creep rate relation may not be expressed accurately by the parabolic type of power function assumed in the usual straight-line log-log plot. When test observations are limited, as in the present case, more conservative stress estimates result from the assumption for constant temperature that a hyperbolic sine relation exists, or that

$$v_0 = v_1 \sinh \frac{\sigma}{\sigma_1} \quad \ldots\ldots\ldots (3)$$

where:

v_0 = minimum creep rate in per cent per hour,

σ = applied stress in pounds per square inch, and

v_1 and σ_1 = constants dependent upon the material and the temperature.

For each of the three test temperatures, the constants v_1 and σ_1 (in Eq. 3) may be evaluated by one of the methods that have been suggested (**29**, **30**), leading to the following relations:

For 838 F. (448 C.)

$$v_0 = 0.117 \times 10^{-5} \sinh \frac{\sigma}{12{,}290}$$

For 998 F. (537 C.)

$$v_0 = 0.035 \times 10^{-5} \sinh \frac{\sigma}{2502}$$

For 1199 F. (647 C.)

$$v_0 = 0.280 \times 10^{-5} \sinh \frac{\sigma}{1356}$$

These are the equations for the three curves representing the test temperatures in Fig. 11 and they permit computation of the ordinates for any desired stresses located on the three known temperature lines of Fig. 12. By using temperature as a parameter in one case and stress in the other, it is possible to interpolate intermediate stress and temperature lines consistent with the known data. It is required that the stress-creep rate relations for the interpolated curves of Fig. 11 follow some simple function of the temperature and that their intersections with lines of constant stress give data for consistent interpolated curves in Fig. 12. This is a trial-and-error procedure resulting in two consistent families of curves covering the desired ranges of stress and temperature. When properly constructed, any combination of stress and temperature will indicate the same creep rate in either Fig. 11 or Fig. 12. It is then possible to tabulate creep rates at any desired stress and temperature intervals as in Table I.

A similar procedure is followed with respect to the intercept ϵ_0. Studies at relatively low temperatures indicate that an assumed hyperbolic sine relation between stress and intercept is reasonable and the following equations may be written as tentative expressions of the effect of stress at three constant temperatures:

For 838 F. (448 C.)

$$\epsilon_0 = 0.0608 \sinh \frac{\sigma}{16{,}310}$$

TABLE I.—CREEP CURVE CONSTANTS—EFFECT OF STRESS AND TEMPERATURE UPON CREEP RATE, v_0. 35 PER CENT NICKEL, 19 PER CENT CHROMIUM ALLOY.

(For use in tangent or asymptote equation ϵ $\epsilon_0 + v_0t$)

Stress, psi.	Creep Rate ($v_0 \times 10^4$)—per cent per hr.[a]								
	800 F. (425 C.)	850 F. (455 C.)	900 F. (480 C.)	950 F. (510 C.)	1000 F. (540 C.)	1050 F. (565 C.)	1100 F. (595 C.)	1150 F. (620 C.)	1200 F. (650 C.)
1 000........	0.00090	0.00098	0.00110	0.00125	0.00145	0.00176	0.00234	0.00395	0.0226
2 000........	0.00178	0.00197	0.00220	0.00253	0.00305	0.00394	0.00573	0.0117	0.0581
3 000........	0.00260	0.00295	0.00340	0.00408	0.00528	0.0074	0.0117	0.0255	0.1266
4 000........	0.00348	0.00403	0.00484	0.00610	0.00840	0.0129	0.0224	0.052	0.2672
5 000........	0.00433	0.00504	0.00640	0.0088	0.0130	0.0220	0.042	0.103	0.5598
6 000........	0.00524	0.00620	0.00820	0.0121	0.0198	0.0367	0.076	0.203	1.171
7 000........	0.00620	0.00740	0.0104	0.0166	0.0297	0.0602	0.137	0.40	2.45
8 000........	0.00721	0.00870	0.0129	0.0225	0.0445	0.100	0.25	0.78	5.12
9 000........	0.00823	0.0101	0.0158	0.0300	0.066	0.164	0.46	1.58	10.71
10 000........	0.00925	0.0117	0.0194	0.0403	0.098	0.270	0.83	3.02	22.40
11 000........	....	....	....	....	0.150	0.42	1.5	....	....
12 000........	....	0.0149	0.0298	0.074	0.224	0.73	2.8	12.0	....
13 000........	....	....	....	....	....	1.15	4.5	....	....
14 000........	....	0.0187	0.0435	0.139	0.490	2.00	8.5	....	....
15 000........	....	....	....	....	....	....	....	....	....
16 000........	....	0.0240	0.070	0.240	1.13	5.40	....	....	....
17 000........	....	....	....	....	....	....	....	....	....
18 000........	....	0.0305	0.103	0.47	2.55	....	....	....	....
19 000........	....	....	....	....	....	....	....	....	....
20 000........	....	0.0375	0.160	0.82	5.70	....	....	....	....
21 000........	....	....	....	....	....	....	....	....	....
22 000........	....	0.0485	0.225	....	....	....	....	....	....
23 000........	....	....	....	....	....	....	....	....	....
24 000........	....	0.0540	0.285	....	....	....	....	....	....
25 000........	....	....	0.330	....	....	....	....	....	....
26 000........	....	0.0660	0.395	....	....	....	....	....	....
27 000........	....	....	....	....	....	....	....	....	....
28 000........	....	0.0770	0.580	....	200	....	....	....	....
29 000........	....	....	....	....	....	....	....	....	....
30 000........	....	0.0980	0.890	....	....	....	....	....	....

[a] Values interpolated from tests at: 838 F. (448 C.)—10,000, 20,000 and 30,000 psi.
998 F. (537 C.)— 5000, 10,000 and 15,000 psi.
1199 F. (648 C.)— 4000 and 7000 psi.

TABLE II.—CREEP CURVE CONSTANTS—EFFECT OF STRESS AND TEMPERATURE UPON CREEP CURVE INTERCEPT, ϵ_0. 35 PER CENT NICKEL, 19 PER CENT CHROMIUM ALLOY.

(For use in tangent or asymptote equation $\epsilon = \epsilon_0 + v_0t$)

Stress psi.	Intercept (ϵ_0), per cent[a]								
	800 F. (425 C.)	850 F. (455 C.)	900 F. (480 C.)	950 F. (510 C.)	1000 F. (540 C.)	1050 F (565 C)	1100 F. (595 C.)	1150 F. (620 C.)	1200 F. (650 C.)
1 000........	0.00421	0.00421	0.00421	0.00422	0.00422	0.00428	0.00464	0.00655	0.0182
2 000........	0.00841	0.00842	0.00843	0.00844	0.00845	0.00870	0.00945	0.0134	0.0370
3 000........	0.0125	0.0126	0.0126	0.0127	0.0127	0.0129	0.0140	0.0213	0.0569
4 000........	0.0167	0.0168	0.0168	0.0169	0.0169	0.0172	0.0191	0.0291	0.0785
5 000........	0.0209	0.0210	0.0211	0.0211	0.0211	0.0218	0.0236	0.0360	0.1026
6 000........	0.0251	0.0252	0.0253	0.0253	0.0253	0.0255	0.0288	0.0443	0.1298
7 000........	0.0293	0.0294	0.0294	0.0299	0.0307	(0.0338)	(0.0428)	(0.0725)	0.1610[b]
8 000........	0.0332	0.0336	0.0345	0.0358	0.0387	(0.0464)	(0.0640)	(0.105)	(0.1972)
9 000........	0.0370	0.0378	0.0394	0.0428	0.0485	(0.0615)	(0.0850)	(0.137)	(0.2394)
10 000........	0.0410	0.0426	0.046	0.0515	0.0606	(0.0815)	(0.114)	(0.173)	(0.2890)
11 000........	....	....	....	....	(0.0606)	....	(0.12)	....	....
12 000........	....	0.050	0.055	0.058	(0.0606)	(0.0815)	(0.13)	....	....
13 000........	....	....	....	....	....	....	(0.14)	....	....
14 000........	....	0.059	0.060	0.0615	(0.0606)	(0.0815)	(0.15)	....	....
15 000........	....	....	....	....	....	....	....	....	....
16 000........	....	0.070	0.066	0.064	(0.0606)	(0.0815)[b]	....	....	....
17 000........	....	....	....	....	....	....	....	....	....
18 000........	....	0.082	0.073	0.067	(0.0606)	(0.0815)	....	....	....
19 000........	....	....	....	....	....	....	....	....	....
20 000........	....	0.095	0.080	0.070	(0.0606)	(0.0815)	....	....	....
21 000........	....	....	....	....	....	....	....	....	....
22 000........	....	....	....	....	....	....	....	....	....
23 000........	....	....	....	....	....	....	....	....	....
24 000........	....	....	....	....	....	....	....	....	....
25 000........	....	....	(0.08)[b]	....	....	....	....	....	....
26 000........	....	....	....	....	....	....	....	....	....
27 000........	....	....	....	....	....	....	....	....	....
28 000........	....	....	....	....	....	....	....	....	....
29 000........	....	....	....	....	....	....	....	....	....
30 000........	....	....	(0.08)	....	....	....	....	....	....

[a] Values interpolated from tests at: 838 F. (448 C.)—10,000, 20,000 and 30,000 psi.
998 F. (537 C.)— 5000, 10,000 and 15,000 psi.
1199 F. (648 C.)— 4000 and 7000 psi.

[b] Bracketed values uncertain because of tendency for ϵ_0 to decrease at high stresses and temperatures.

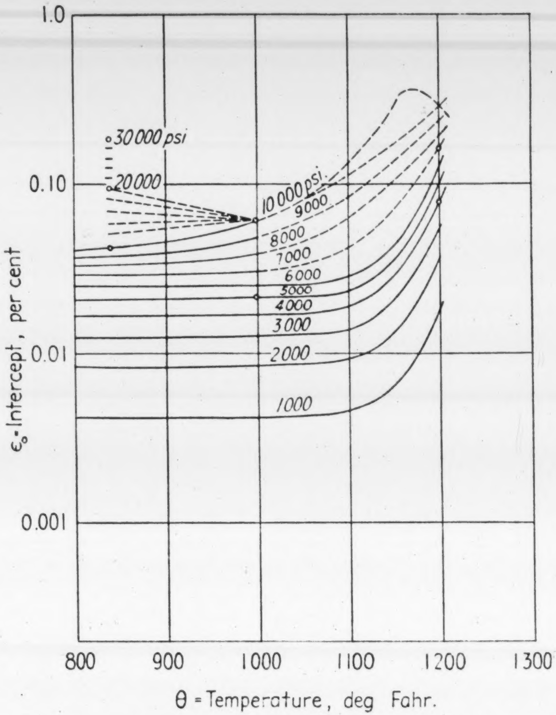

Fig. 13.—Temperature-Intercept-Stress Relations. From tension creep tests of 35 per cent nickel, 19 per cent chromium alloy.

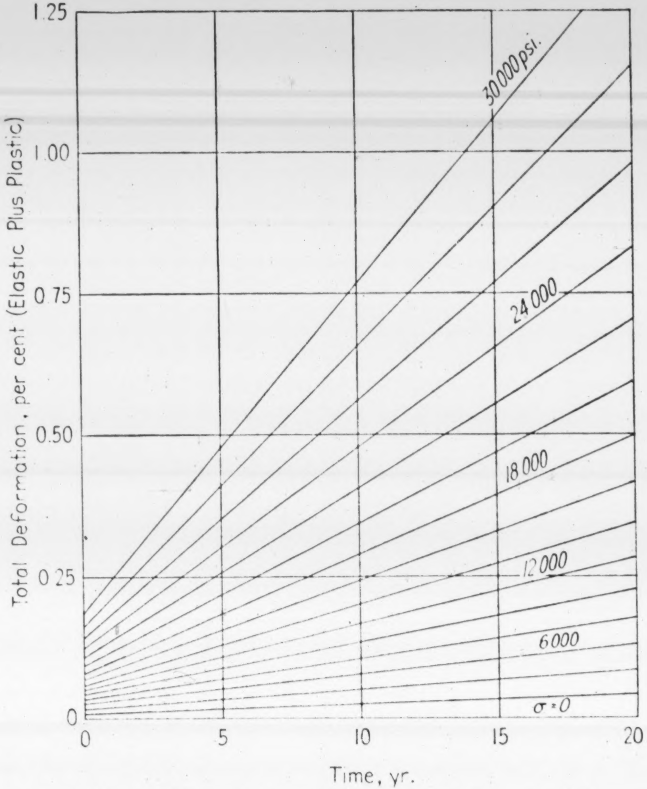

Fig. 14.—Interpolated and Extrapolated Creep Curves. For 35 per cent nickel, 19 per cent chromium alloy at 838 F. (448 C.).

For 998 F. (537 C.)

$$\epsilon_0 = 0.0143 \sinh \frac{\sigma}{4650}$$

For 1199 F. (648 C.)

$$\epsilon_0 = 0.1033 \sinh \frac{\sigma}{5706}$$

Recent studies at 1200 F. (650 C.) and other available data indicate that the intercept reaches a maximum as the stress is increased and then decreases. This requires a departure at high stresses and temperatures from the assumed hyperbolic sine relations. The region in which the relations are uncertain is indicated by dotted curves in Fig. 13. It is quite possible that one or both of the test observations at 1199 F. (648 C.) lie on a decreasing branch of the curve. More tests would be necessary to clarify this situation. Fortunately, the importance of the intercept decreases as the stress and temperature are increased. For this reason it is not necessary to defer analysis of test data until more is known about the dotted portion of the curves.

When Fig. 13 has been constructed to represent the best available chart of variations of ϵ_0 with stress and temperature, it becomes possible to pick off and tabulate intercepts at any desired stress and temperature intervals as shown in Table II. The correlation and tabulation or charting of values of the creep rate v_0 and the intercept ϵ_0 over the ranges of stress and temperature studied are recommended as useful methods of presentation of creep data for subsequent use.

The estimation of working stresses and charts for comparison of materials are simple problems when the values for intercept ϵ_0 and creep rate v_0 are known for various stresses at the service temperature. If now we agree that the creep curve may be extrapolated with respect to time along the final tangent or asymptote, we may construct as in Fig. 14 a family of such extrapolated curves. A maximum total deformation of 1.25 per cent and a maximum time of 20 yr. are shown, but any desired limit of deformation and expected service life may be chosen and a chart drawn at any temperature covered in Figs. 11, 12, and 13 or Tables I and II. It is desirable to construct at intervals several of these charts covering the entire temperature range.

The next requirement is some statement of the criterion governing the choice of working stresses or stress estimates for comparison of materials. This may be any allowable deformation within any specified time such as 0.1, 0.5, or 1.0 per cent in 2000, 10,000, or 100,000 hr., chosen to suit the intended design applications. This combination of deformation and time is represented as the point *B* in Fig. 7. If, for example, we wish the stress at 838 F. (448 C.) estimated to produce 0.2 per cent total deformation in 10 yr., Fig. 14 shows it to be approximately 14,000 psi.

Using the same method on charts for other temperatures, a curve may be drawn representing the stress-temperature relations for any desired combination of allowable deformation and expected service life. Figure 15 represents various deformations at the end of a given time, and Fig. 16 represents various times to reach a given total deformation. Any desired criterion for comparison of stress-temperature relations may be taken from Figs. 15 or 16 and plotted for comparison with similar curves for other alloys. Figure 17 shows the appearance of such a chart. Charts of these types are recommended for comparison of materials and for choice of working stresses.

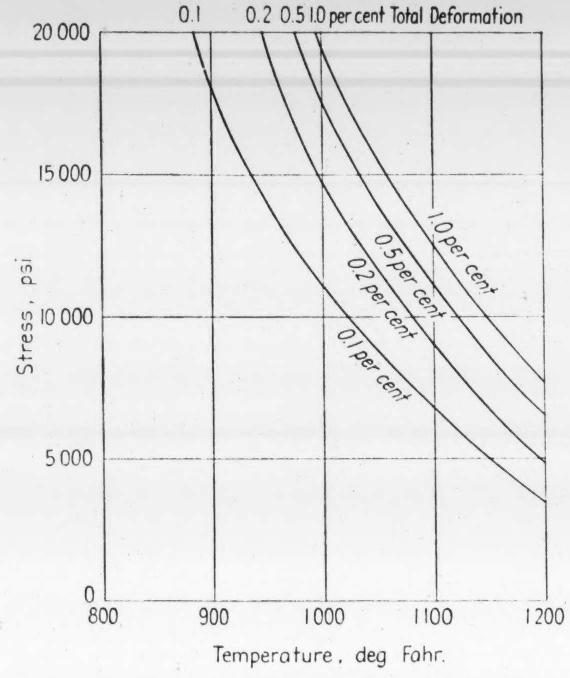

Fig. 15.—Stress-Temperature Relations For Various Deformations at End of 2000 hr., 35 per cent nickel, 19 per cent chromium alloy.

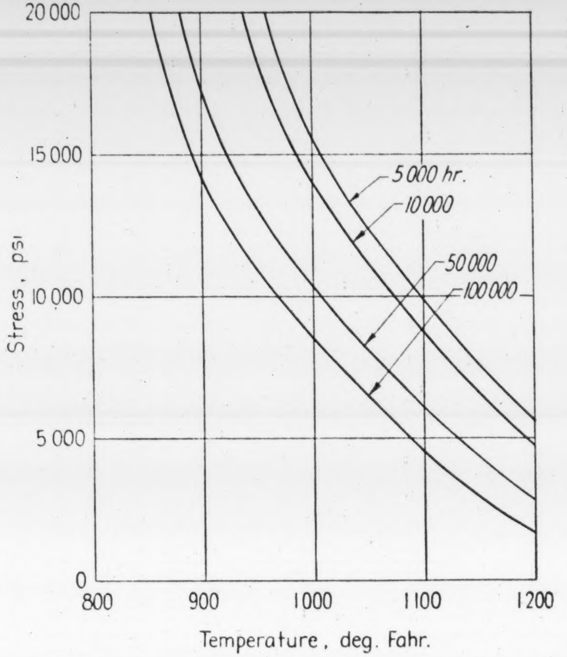

Fig. 16.—Stress-Temperature Relations For 0.5 per cent Total Deformation at End of Various Times, 35 per cent nickel, 19 per cent chromium alloy.

It is obvious that the relative merits of different materials will depend somewhat upon the criterion used. When these rough strength comparisons, based on a minimum number of tests, indicate the most desirable alloys for a given application, other characteristics may be studied in choosing the alloys for which further tests are needed. These tests can then be made and the analysis repeated using the additional data. In this way, more reliable working stress values may be obtained.

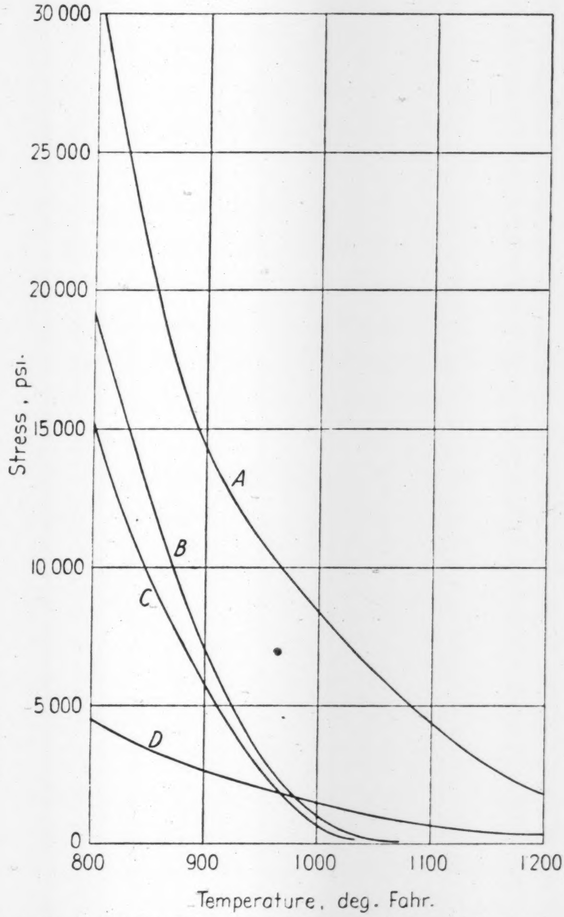

Fig. 17.—Comparison of Materials. For 0.5 per cent total deformation in 100,000 hr.

Conclusions

It is expected that any form of interpretation of this kind will require revision from time to time. Some assumptions are necessary to reduce the number and duration of tests which might permit more reliable analysis. Until more information is available, it is felt that tentative acceptance of some systematic form of interpretation can be used to correlate existing test data at various stresses and temperatures from different laboratories as well as to compare and utilize the data being collected for a large number of new alloys. This correlation will show up immediately the ranges of stress and temperature in which further tests are necessary to give needed information. When this background of known information is established, the evaluation of a new alloy may be accomplished with a minimum number of tests and its comparative value fixed with reasonable accuracy. The advantage claimed for the proposed method is that it permits a reasonably accurate analysis based on an absolute minimum number of tests. This can be corrected to give more reliable results when more test data become available. It is hoped that this advantage will outweigh the objections to extrapolation and other assumptions necessary to effect a comparison of materials and to present a reasonable basis for the choice of working stresses.

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DISCUSSION

Mr. R. G. Sturm[1] *(presented in written form).*—The author is to be congratulated for presenting a clear and open-minded discussion of an interpretation of creep data.

Quite frequently creep is referred to as an undesirable phenomenon but when the effects of creep are considered from several points of view, it is found that for each case in which creep is disadvantageous there is some other case in which creep is definitely advantageous.

In general, the disadvantages of creep may be summarized as: (1) continued deformation which exceeds permissible tolerances and thereby renders a machine, apparatus, or structural part inoperative; (2) continued deformation of machine or structural parts which have a tendency to become unstable with excessive deformation, which parts may fail by buckling as a result of continued creep.

To offset these two general disadvantages of creep are the following advantages: (1) creep deformations at points of highly localized stress tend to redistribute the stresses and delay or prevent failure; (2) in some cases, such as internal pressure vessels with dished heads, the deformations of the part under load are such that the load-carrying capacity of the member is increased by virtue of deformations resulting from creep.

We agree wholeheartedly with the author in his statement that "To secure the most effective utilization of creep data, it is important that methods of interpretation be reviewed occasionally to follow changing concepts of high temperature problems." This is equally true of room temperature problems. While the author's method of interpretation may apply to certain uses of steels at elevated temperatures, difficulties have been encountered in trying to apply the concept of creep rate to the interpretation of creep data on aluminum alloys.

It is interesting to note that the disadvantages and advantages resulting from creep depend upon the total amount of creep that occurs. In view of this fact, it would seem that the method of interpreting creep data which will yield information about the total creep occurring for any period of time would be the most generally applicable method.

It has been noted that a number of investigators consistently use the creep rate as the criterion for interpreting creep data. If a material flows in a manner similar to that of a truly plastic material, such that the time-deformation curve is a straight line, the creep rate multiplied by the elapsed time will give a measure of the total creep during that period. When the time-deformation curve is not a straight line, however, the creep rate is continuously changing and conclusions drawn from any arbitrarily chosen value may be quite misleading.

To illustrate the difficulties encountered in using creep rates, actual creep data obtained at room temperature on aluminum wire of electrical conductor

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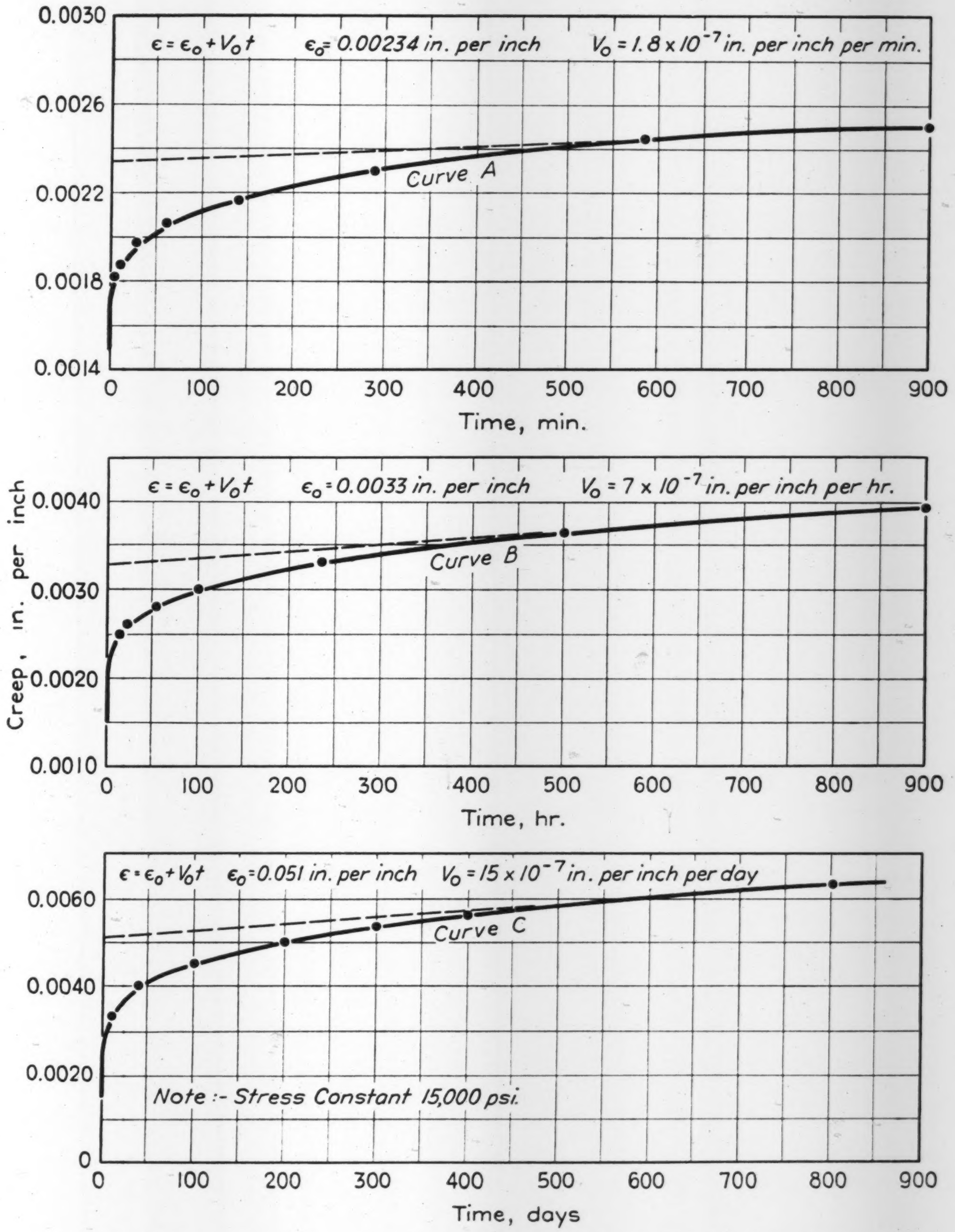

FIG. 1.—Creep Data at Room Temperatures for Aluminum Wire of Electrical Conductor Grade.

0.149 in. in diameter, 100 in. in gage length.

grade at a constant tensile stress of 15,000 psi. have been plotted in the accompanying Fig. 1. Curve *A* has been plotted to a rather expanded horizontal scale. As nearly as can be detected by observing the curve, the curve becomes a straight line at the right-hand side of the plot. In applying the method suggested by the author to this, an equation is obtained for the tangential straight line. This line, of course, is not an asymptote.

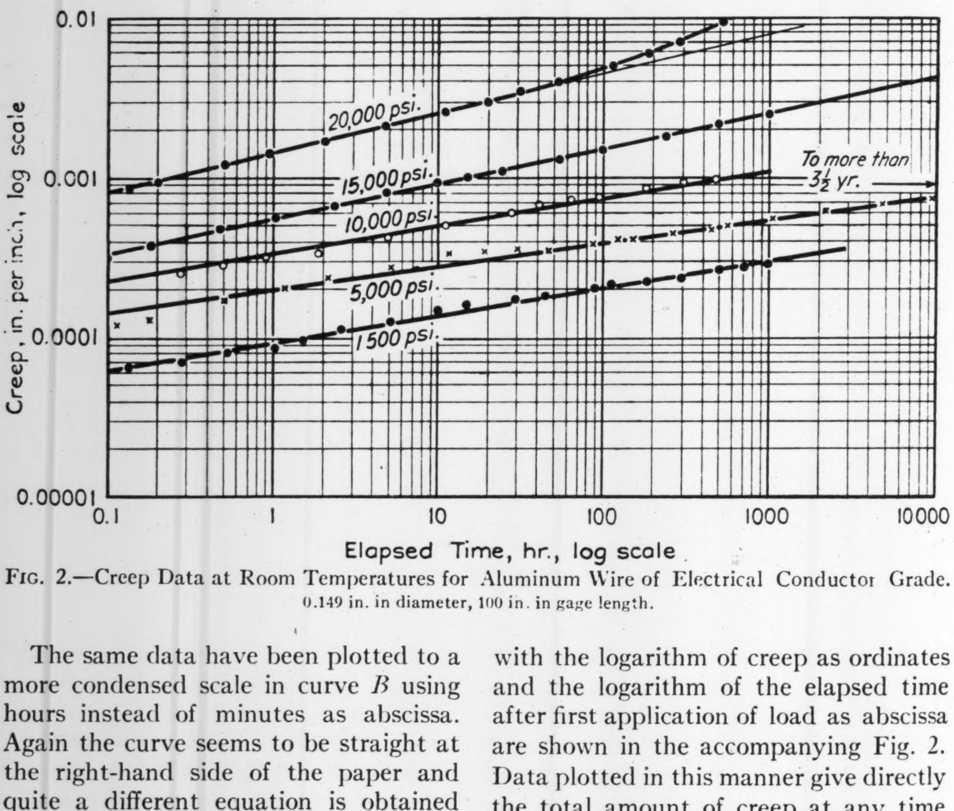

FIG. 2.—Creep Data at Room Temperatures for Aluminum Wire of Electrical Conductor Grade. 0.149 in. in diameter, 100 in. in gage length.

The same data have been plotted to a more condensed scale in curve *B* using hours instead of minutes as abscissa. Again the curve seems to be straight at the right-hand side of the paper and quite a different equation is obtained for the straight line. When the data are plotted to a still more condensed scale on the abscissa, curve *C*, that is, using days instead of hours, a third equation is obtained. Therefore, one is forced to determine arbitrarily some horizontal scale and some vertical scale and say the data must be plotted to those scales in order to obtain comparable values. Under such conditions one would not expect reliable interpretations for widely different materials.

The difficulties encountered in creep rate have been recognized for a number of years and a method of considering the actual creep data plotted on log-log paper was proposed in 1936,[2] and subsequently used in a recent analytical paper.[3] The data shown in Fig. 1, when plotted with the logarithm of creep as ordinates and the logarithm of the elapsed time after first application of load as abscissa are shown in the accompanying Fig. 2. Data plotted in this manner give directly the total amount of creep at any time for the applied stress. With relatively few curves it is possible to estimate the creep for any stress at any time. When the desirable life of the part under consideration and the permissible tolerances in deformation are set, the maximum stress that will not produce creep in excess of the tolerances in a time equal to the desired life may be determined directly from the data. If adjustments are made after a period of loading, creep subsequent to these adjustments can

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[2] R. G. Sturm, C. Dumont, and F. M. Howell, "A Method of Analyzing Creep Data," *Journal of Applied Mechanics*, Am. Soc. Mechanical Engrs., Vol. 3, No. 2, June, 1936, pp. A-62-66.

[3] Figure 7 of the paper by Evan A. Davis, "Creep and Relaxation of Oxygen-Free Copper," *Journal of Applied Mechanics*, Vol. 10, No. 2, June, 1943, pp. A-101-105.

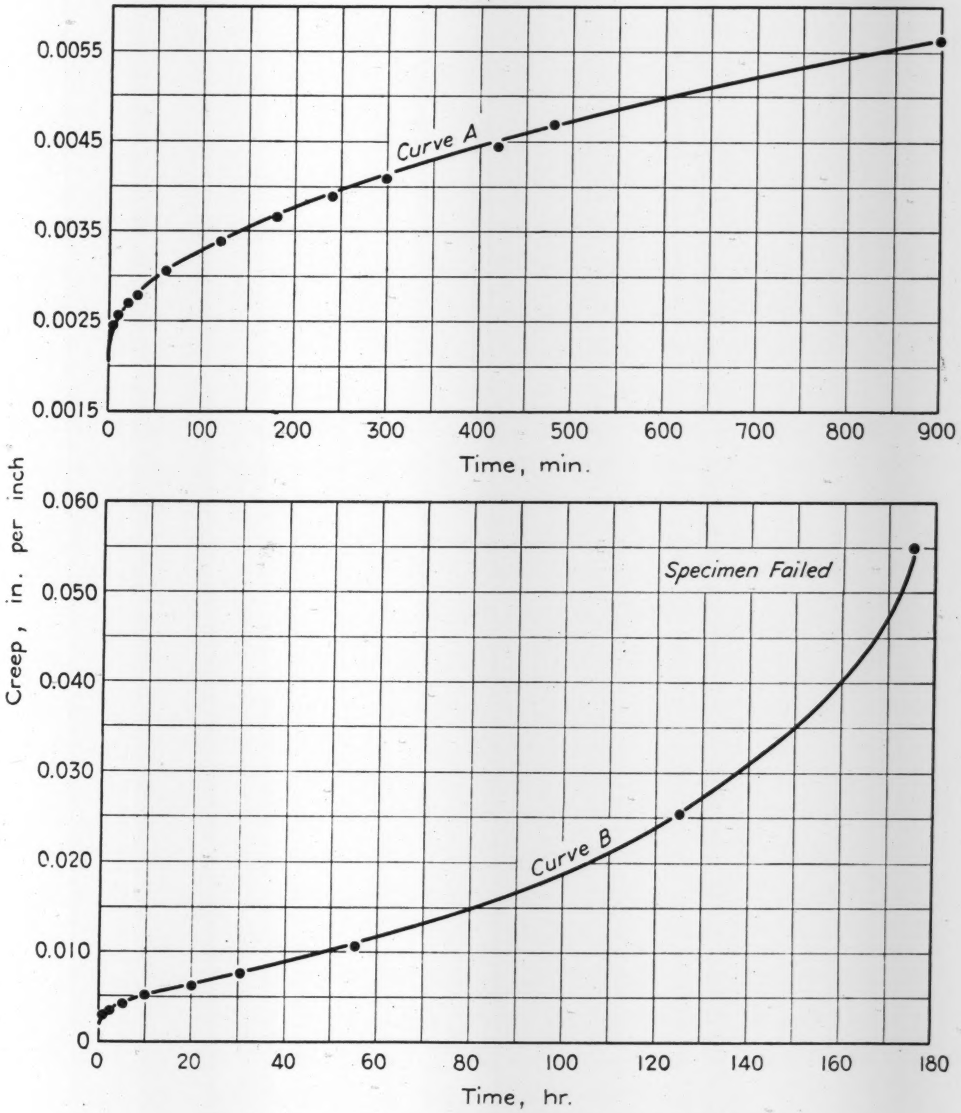

FIG. 3.—Creep Data at 400 F. (205 C.) for 18S-T Rod.
0.75 in. in diameter, 2 in. in gage length.

readily be determined as the difference in total creep before and after the adjustments.

In some cases, especially overhead transmission lines, it may be desirable to prestress the member (in this case the cable) for a period of a few minutes at a stress considerably above the operating stress. Test data have borne out the fact that the total deformation at the end of a short prestressing time can be used as a basis for determining the length of time during which the member (cable) will deform not more than that amount at a lower stress. This procedure has actually been followed in the erection of some overhead transmission lines.

Short-time static overloading of parts containing unavoidable stress concentrations has been reported to increase their life in fatigue. Adequate creep data, giving total creep *versus* time, can be very useful in estimating the time and stress for such preloadings.

Difficulty in using creep rate has also been encountered in trying to interpret creep at elevated temperatures. In the accompanying Fig. 3 are plotted creep data obtained at 400 F. (205 C.) for a heat-treated wrought aluminum alloy at a stress of 20,000 psi. Curve *A* shows the time and creep scales expanded, whereas curve *B* shows the same data to a more condensed scale. These data, together with other data, are shown also in the accompanying Fig. 4.

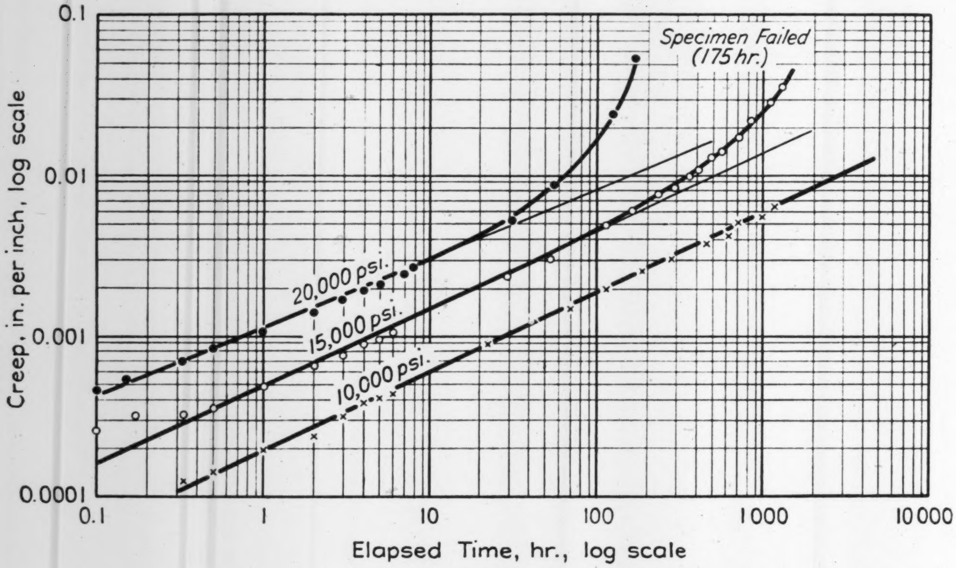

FIG. 4.—Creep Data at 400 F. (205 C.) for 18S-T Rod.
0.75 in. in diameter, 2 in. in gage length.

The log-log plot of creep-time data not only lends itself to ready interpolation and extrapolation but also serves as a warning of impending failure long before failure occurs. In Fig. 4 the two upper curves deviate from the straight lines and curve upward. Many tests have shown that when the curve starts to turn upward failure will follow.

Our records show that in one case ultimate failure of a test specimen was predicted to occur several years later, and it did fail in due course of time. Of course stresses high enough to cause fracture in a reasonably short time are not intentionally used in service, but with the log-log plot of creep *versus* time at stresses which are generally encountered in service an estimate can be made of the service life that can be expected before creep may threaten the stable load-carrying capacity of the member, or before the deformation tolerances permitted by the construction are exceeded. Such plots may also serve as a basis for estimating the time and temperature for stress relieving.

MR. RICHARD F. MILLER.[4]—In the type of creep test discussed in this paper, the load is applied in one rather brief operation. I should like to ask Mr. McVetty for his opinion of the effect on creep behavior of different rates of loading.

MR. P. G. MCVETTY[5] (*author's closure, by letter*).—In reply to Mr. Miller's question, the application of load at high temperature causes an elastic deformation proportional to the stress and a plastic deformation which increases with stress and time. Changes in rate of loading produce different amounts of deformation prior to the constant-stress test. It is customary to assume that this plastic deformation is a part of the total creep and it is added as a correction. We have found it desirable to reduce the loading time to a minimum so that practically all of the measured creep occurs at the specified constant stress. With this procedure, the correction for variable stress is small and the possible effects of loading procedure upon subsequent creep behavior are reduced to a minimum.

Mr. Sturm recommends a method of interpretation which he has used in the application of aluminum and its alloys at normal and slightly elevated temperatures. He suggests extension of its use to high-temperature applications of heat-resisting alloys. This method eliminates the conventional concept in which the minimum creep rate is considered a significant constant. It assumes, instead, that the creep rate decreases continuously, approaching zero as a limit. This assumption is implied in a straight-line extrapolation of a double-logarithmic plot of the creep-time curve. A decrease in creep rate of this type might be expected to result from strain hardening when annealing effects at the service temperature are negligible.

As the temperature increases, annealing effects tend to increase the creep rate, leading ultimately to failure as shown in Mr. Sturm's Fig. 3. A study of curves of the type shown in Fig. 2 of the paper indicates that the tangent at the inflection point very nearly coincides with the curve over a period of time which increases as the stress decreases. It was shown in Fig. 5 of the paper that the time at nearly constant rate must be long at the low creep rates associated with actual working stresses. During this time, the opposing effects of strain hardening and annealing are approximately balanced. For a wide variety of applications, the assumption that creep rate is constant during the major portion of the life of the material in service has led to a satisfactory choice of working stresses.

Mr. Sturm shows in his Fig. 1 a gradual decrease in creep rate over a period of more than two years. For this type of creep curve, the author proposed in 1933 that extrapolation follow the asymptote rather than the final tangent. This assumes that the creep rate approaches a minimum value which is a function of stress and temperature instead of Mr. Sturm's assumption that it always approaches zero as a limit. A simple, graphical method of determining the minimum value from tests of reasonable duration is indicated in Fig. 3 of the paper. This provides a method applicable to cases in which strain hardening predominates during the test without eliminating the possibility that annealing effects may become important after long exposure to the service temperature. It permits a gradual change in the combined effects of these opposing forces as the temperature increases, without change in the method of interpretation. It is more conservative than an assumption that the creep rate continues to decrease indefinitely, but it avoids extrapolation of the final tangent to the creep curve while the creep rate is still decreasing.

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HYPERBOLIC SINE CHART FOR ESTIMATING WORKING STRESSES OF ALLOYS AT ELEVATED TEMPERATURES

By A. Nadai[1] and P. G. McVetty[1]

Synopsis

Long-time creep tests as the basis for the comparison of existing and new alloys for high-temperature applications require a careful correlation of the test results. To reduce the time required for testing and for determining the creep properties of alloys, creep tests are conducted at stresses higher than those used in service. In this paper a method of plotting results of creep tests in a new manner is suggested which departs from the usual double logarithmic plots of stress and minimum creep rate. The method is based on the hyperbolic sine function for expressing the dependence of the rate of flow on the stress. The new chart is described and it is shown how easily it can be constructed from one single curve. Examples illustrate its application to a variety of materials to obtain estimates of stress for specified low creep rates.

One of the principal factors which affects the choice of safe working stresses from long-time creep tests at the maximum operating temperature is the functional relation between stress and minimum creep rate. In this paper, two new charts are described which have been constructed for facilitating estimates of stress at very low creep rates. One of these two charts is introduced merely for describing certain properties of the second main curve sheet and the intention is to recommend use of the second chart only for the extrapolation of the observed data from test results.

Usually a series of creep tests is run under constant loads at a given temperature. If the permanent portion of the strain (creep) ϵ is plotted as a function of the time t at various loads, a family of curves is obtained similar to the one shown in Fig. 1, each curve having an inflection point. For the practical applications of long-time creep test data it is sufficient and has become customary to substitute for the creep curves $\epsilon = f(t)$ the family of straight lines which are tangent to the creep curves at the inflection points. The following considerations refer to a determination of the slopes of these strain lines for given stresses σ. The family of these straight lines $\epsilon = \epsilon_0 + v\,t$ depends on *two* parameters: the intercept ϵ_0 for $t = 0$ and the slope $v = \dfrac{d\epsilon}{dt}$ of the line. For stable alloys both of these parameters seem to depend on the stress σ. It is beyond the scope of this paper to discuss the relative importance under various conditions of the intercepts ϵ_0 and of the slopes v in order to estimate the total deformation at the end of the time representing the expected service life, t. The dependence of ϵ_0 on the stress σ will not be investigated and with regard to it reference is made to a paper by one of the authors.[2]

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We shall assume that the minimum creep rates $v = \frac{d\epsilon}{dt}$ (ϵ = creep or permanent strain) have been determined by experiments at a series of values of the stress σ.[3]

In order to submit the information for the design engineer who usually is interested in much lower values of the stresses, than the ones under which the creep tests were run, the testing engineer encounters the following problem: suppose that at least two pairs of values v_1, σ_1 and v_2, σ_2 are known. Find a curve or a function which will express the stress σ as a function of the values of an assumed "*permissible*" minimum creep rate v under the service conditions.

This extrapolation problem has been recently discussed in two papers.[4] It was stated that for comparatively large values of the two fundamental variables v and σ an exponential function

$$v = \frac{v_0}{2} e^{\frac{\sigma}{\sigma_0}} \quad \ldots\ldots\ldots(1)^*$$

expresses sufficiently the dependence of v on σ where v_0 and σ_0 are two material constants of the dimensions of a rate of strain and of a stress, which have to be determined from the long-time creep tests. This function fails, however, for small values of σ since it would furnish for $\sigma = 0$, $v = v_0/2$ or a finite creep rate at zero stress or when the bar is unstressed. The same law is expressed also by

$$\sigma = \sigma_0 \log_e \left(\frac{2v}{v_0}\right) \ldots\ldots(2)$$

and in this latter form is known as the

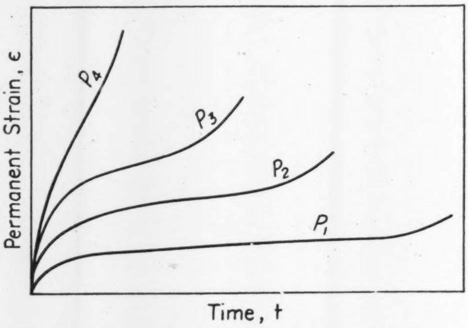

FIG. 1.—Long Time Creep Curves.—Observed Under Constant Loads.

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[2] P. G. McVetty, "Interpretation of Creep Test Data," see p. 707.

[3] When speaking of creep ϵ or of creep rates $\frac{d\epsilon}{dt}$ it should be noted that these terms need a more precise definition than the one which is usually accepted. In a standard long-time tension creep test the load is kept constant. When the length of the test bar increases the cross-sectional area of the bar decreases and consequently the *true* stress in the bar gradually must increase. "Creep rates" $d\epsilon/dt$ are usually referred to the permanent portions of the conventional strains ϵ. What is recorded in the test is the sum of an elastic and a permanent deformation (extension). Since the true stress increases as the bar stretches the elastic portion of the conventional strain must also increase. When computing the permanent portions of the strains, this should be taken into consideration, after assuming that the elastic portions can be taken to be proportional to the *true* stresses. It must be noted that a constant load test is not a constant stress test if the bar stretches by finite amounts. Second, that the conventional rates of strain are not exact measures of the changes in dimensions which take place.

When creep tests are extended to fracture, particularly at higher temperatures, the definitions for strain and rate of strain should be based on more satisfactory variables than ϵ and $v = d\epsilon/dt$, namely on the natural strains $\bar{\epsilon} = \log_e (1 + \epsilon)$. $\bar{\epsilon}$ and the rate $d\bar{\epsilon}/dt$ should not be referred to the stresses σ which are obtained by dividing the loads P by the original area of the cross-section A, but to the true stresses $\bar{\sigma}$ referred to the actual areas $\bar{A}$.

When the symbol $v = d\epsilon/dt$ is used in this paper, it is reserved for designating the minimum rate of strain assuming that the permanent portion of the strain during a test is of such a small magnitude that the changes in the cross-sectional areas of the tensile specimens can be neglected. The dimension of a rate of strain is the reciprocal of a time, for example $v = d\epsilon/dt = 0.0001$ (1/hr.) (the bar stretches permanently at a rate of one ten thousandth of its original length per hour). Speaking of creep rates in terms of "inch per inch per hour" is a pleonasm which can and should be avoided since the linear dimensions cancel out.

[4] P. G. McVetty, "Creep of Metals at Elevated Temperatures. The Hyperbolic Sine Relation Between Stress and Creep Rate," Am. Soc. Mechanical Engrs., Spring Meeting, Davenport, Iowa, April 27, 1943.

P. G. McVetty, "Interpretation of Creep Test Data," see p. 707.

[*] The reason for using $v_0/2$ for one of the constants will be explained later.

logarithmic speed law in plastic deformation or flow. It was described as early as 1909 by Ludwik.[5]

Another much used function expresses the stress σ as a power function[6] of the rate of creep v, which can conveniently be written in the form:

$$\frac{v}{v_0} = \left(\frac{\sigma}{\sigma_0}\right)^m \quad \ldots\ldots\ldots (3)$$

m, σ_0 and v_0 are constants, one of which (either σ_0 or v_0) can arbitrarily be selected. The exponent m is $1 < m < \infty$. The power function introduces certain difficulties,[7] when used for extrapolation to low values of stress or over a wide range of the rates of flow. Experimental evidence available is indicating a systematic deviation of test results from the straight lines at low creep rates which should appear on double logarithmic plots of σ and v corresponding to Eq. 3. It has also been pointed out that theoretical and experimental evidence requires a speed law expression for the stress σ which is an odd function of the rate of flow v, similarly as the law for the flow of a viscous substance states that the rate of shear $d\gamma/dt = \frac{\tau}{\eta}$ is a linear function of the shearing stress τ (η = coefficient of viscosity). If the direction of the shearing stress is reversed, a material must flow with the same rate of shear in the opposite direction. This condition is only satisfied by Eq. 3 if $m = 1, 3, 5, \ldots$, but not for fractional, irrational, or even exponents m. Another defect of Eq. 3 is that the viscosity $d\sigma/dv$ becomes infinite at $v = 0$, $\sigma = 0$ if $1 < m < \infty$ which is an objectionable conclusion from a physical viewpoint. However, the power function is useful in the development of exact expressions for the creep of metals under combined stress, which have interesting practical applications.[8]

The above-mentioned difficulties can be avoided by assuming for the creep speed law the hyperbolic sine function:

$$v = v_0 \sinh \frac{\sigma}{\sigma_0} \quad \ldots\ldots\ldots (4)$$

v_0 and σ_0 designate two material constants of the dimensions of a rate of strain and of a stress. Equation 4 has been used with good results by one of the authors since 1933 for plotting creep and faster constant strain rate tests at elevated temperatures in various practical applications at the Westinghouse Research Laboratories.[9]

The hyperbolic sine function receives support from the logarithmic speed law of Ludwik,[5] the mechanical model of a solid which was suggested by Prandtl,[10] by various experimental investigations on plastic flow, and by the chemical reaction rate theory by Eyring.[11] and his associates.

Since the hyperbolic sine function

$$v = v_0 \sinh \frac{\sigma}{\sigma_0} = \frac{v_0}{2}\left(e^{\frac{\sigma}{\sigma_0}} - e^{-\frac{\sigma}{\sigma_0}}\right) .. (5)$$

for large values of σ/σ_0 approaches the exponential function expressed by the first term in parentheses, we note that for comparatively large values of the stresses, Eq. 5 becomes identical with Eqs. 1 and 2 expressing the logarithmic speed law. For small values of σ or v the function Eq. 5 becomes $v = v_0\sigma/\sigma_0$ defining a finite viscosity $\eta = \sigma_0/v_0$ of the material at zero stress.

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[5] P. Ludwik, "Elemente der Technologischen Mechanik," Julius Springer (Berlin), p. 47 (1909).

[6] F. H. Norton, "The Creep of Steel at High Temperatures," McGraw-Hill Book Co., Inc., New York, N. Y. (1929).

[7] P. G. McVetty, "Creep of Metals at Elevated Temperatures. The Hyperbolic Sine Relation Between Stress and Creep Rate," Am. Soc. Mechanical Engrs., Spring Meeting, Davenport, Iowa, April 27, 1943.

[8] A. Nadai, "On the Creep of Solids at Elevated Temperatures," *Journal of Applied Physics*, Vol. 8, No. 6. June, 1937, p. 418.

[9] A. Nadai, "The Influence of Time Upon Creep. The Hyperbolic Sine Creep Law," Stephen Timoshenko Anniversary Volume, The Macmillan Co., New York, N. Y., p. 155 (1938).

[10] L. Prandtl, "Ein Gedankenmodell zür kinetischen Theorie der festen Körper," *Zeitschrift für angewandte Mathematik und Mechanik*, Vol. 8, pp. 85–106 (1928).

[11] S. Glasstone, K. J. Laidner and H. Eyring, "The Theory of Rate Processes," McGraw-Hill Book Co., Inc., New York, N. Y. (1941).

H. Eyring, "Viscosity, Plasticity and Diffusion as Examples of Absolute Reaction Rates," *Journal of Chemical Physics*, Vol. 4, pp. 283–291, April (1936).

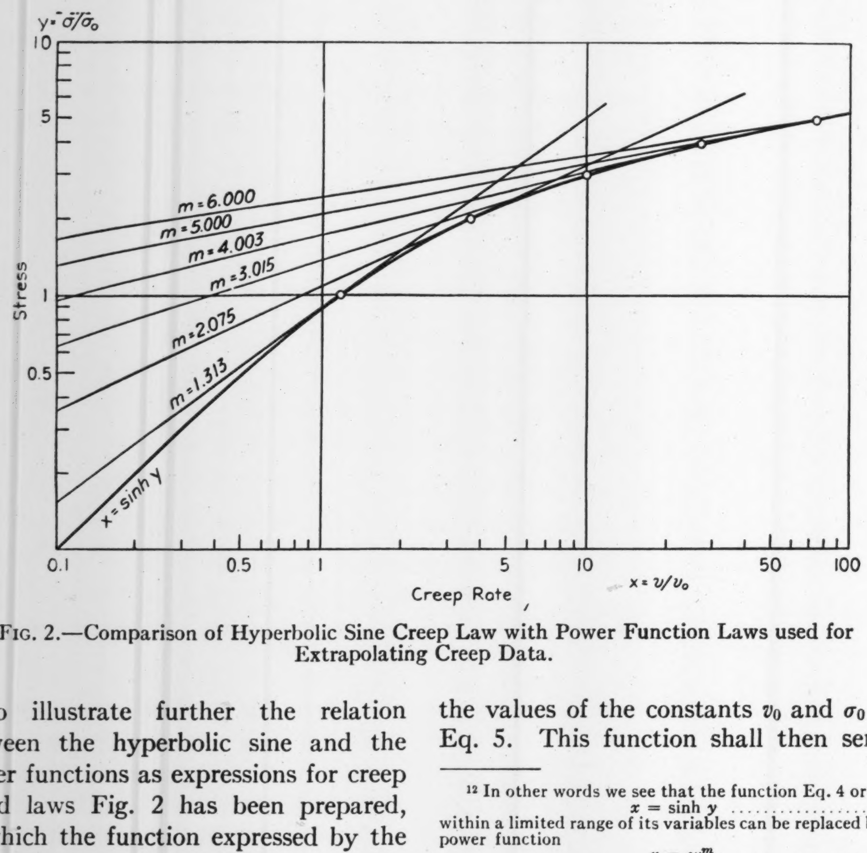

FIG. 2.—Comparison of Hyperbolic Sine Creep Law with Power Function Laws used for Extrapolating Creep Data.

To illustrate further the relation between the hyperbolic sine and the power functions as expressions for creep speed laws Fig. 2 has been prepared, in which the function expressed by the Eqs. 4 or 5 has been plotted on double logarithmic paper by using

$$x = \frac{v}{v_0}, \qquad y = \frac{\sigma}{\sigma_0}$$

as the variables. The curve drawn with a heavy line in Fig. 2 represents Eq. 4 or 5. It is noted that this curve for decreasing values of x and y becomes tangent to a straight line having the slope 1. Any tangent drawn to this curve represents the osculating power function in the vicinity of the point of tangency.[12]

The problem of finding a suitable expression or curve for extrapolation of creep observations can now be restated as follows: given a pair of creep observations (v_1, σ_1) and (v_2, σ_2) one shall find the values of the constants v_0 and σ_0 in Eq. 5. This function shall then serve

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[12] In other words we see that the function Eq. 4 or

$$x = \sinh y \quad \ldots\ldots\ldots\ldots\ldots\ldots (6)$$

within a limited range of its variables can be replaced by a power function

$$x = cy^m \quad \ldots\ldots\ldots\ldots\ldots\ldots (7)$$

by drawing the tangent in Fig. 2 to the curve representing Eq. 6 after properly determining the constants c and m. The straight line corresponding to Eq. 7 in Fig. 2 becomes tangent to the curve representing Eq. 6 in a point x_0, y_0 if one chooses for

$$m = y_0 \operatorname{cotan} y_0,\ c = \frac{\sinh y_0}{y_0{}^m} \quad \ldots\ldots\ldots\ldots (8)$$

This has been shown in a number of points in Fig. 2 and the corresponding power function exponents m were indicated along the tangents to the curve representing the hyperbolic sine function in the double logarithmic plot. The common points were

$y_0 = 1$	2	3	4	5	6
$x_0 = 1.175$	3.627	10.02	27.29	74.20	201.7

the corresponding values of the exponents m computed from Eq. 8 were:

$m = 1.313$	2.075	3.015	4.003	5.000	6.000

The exponents m of the osculating power functions approach rapidly the value of y_0 with increasing values of the ordinates of the points of tangency. When $y_0 > 3$ practically $m = y_0$ expressing a well-known property of an exponential function, namely that its slope is equal to its ordinate.

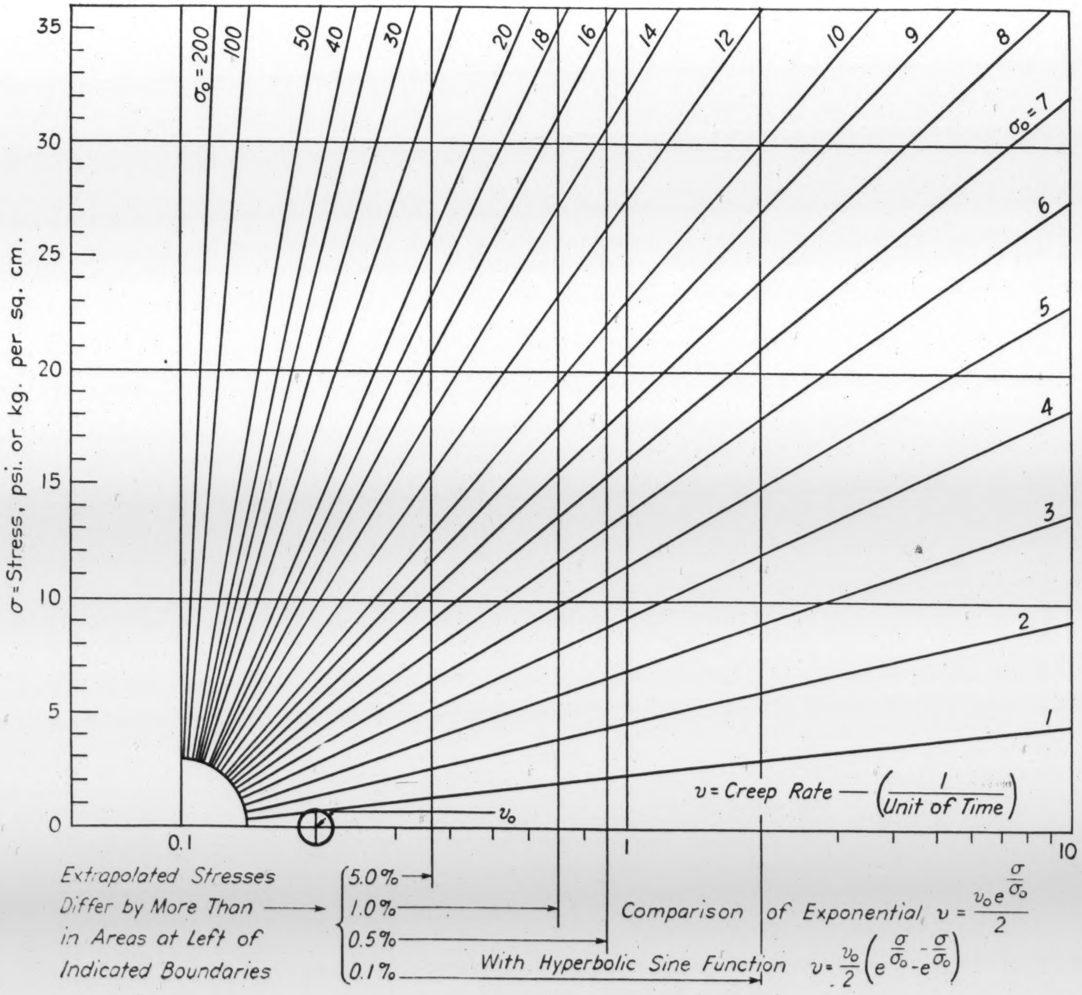

FIG. 3.—Chart for Determining Constants, v_0, and σ_0 in Exponential Function Between Stress — σ and Creep Rate — v.

$$v = \frac{v_0 e^{\frac{\sigma}{\sigma_0}}}{2}$$

to define corresponding values of v and σ in the range of small values of these two variables.

This requires finding the roots v_0 and σ_0 common to the two equations:

$$v_1 = v_0 \sinh \frac{\sigma_1}{\sigma_0}, \qquad v_2 = v_0 \sinh \frac{\sigma_2}{\sigma_0} \,..\, (9)$$

when v_1, v_2, σ_1, σ_2 are given constants. If *either* the parameter v_0 *or* the parameter σ_0 is varied, an equation like

$$\frac{v}{v_0} = \sinh \frac{\sigma}{\sigma_0} \,........\, (10)$$

represents two families of curves assuming that v and σ are rectangular running coordinates.[7]

For the following it appears convenient *to study these two families of curves in a semilogarithmic plot*, in which σ is plotted vertically on ordinary scale and v is plotted horizontally on a logarithmic scale. Due to the fact that the two constant parameters v_0 and σ_0 in Eq. 10 appear only in the combinations v/v_0 and σ/σ_0 all curves in the two families can be constructed in the simplest manner from one single curve, whose points have been computed.

This must also be true for the families of curves (Eq. 2)

$$\frac{\sigma}{\sigma_0} = \log_e \frac{v}{\left(\frac{v_0}{2}\right)} \,.......\, (11)$$

expressing the logarithmic speed law. If the parameter σ_0 is varied Eq. 11 represents a pencil of rays emanating from the point $v = v_0/2$ on the abscissa axis. This family of straight lines is indicated in Fig. 3 representing the logarithmic law in the chart. Around the point of convergence of these lines a large circle is drawn assuming for $v_0/2 = 0.1$ (1/hr.) or $v_0 = 0.2$ (1/hr.). The point $v_0 = 0.2$ is indicated by a smaller circle (target) and along some of these straight lines the corresponding values of σ_0 are shown. Changing to another value of the parameter v_0 simply means shifting of the pencil of rays to a new position in the horizontal direction. Consequently, if two points (v_1, σ_1) and (v_2, σ_2) for the function Eq. 11 are prescribed, the unknown values of the parameters v_0 and σ_0 must be found by drawing the straight line connecting these two points (on the semilogarithmic chart). The intercept of this line on the abscissa axis furnishes the value $v_0/2$ and the slope determines the value of σ_0, which can also be read on the straight line parallel to it in the family of straight lines.

Returning now to the hyperbolic sine functions in the semilogarithmic representation, since these functions for sufficiently large values of the variables v or σ must approach the function expressed by Eq. 11 we see that the straight lines in the first chart must represent the asymptotes of the corresponding curves in the second chart.

In Fig. 4 (Plate IV) these curves, representing the hyperbolic sine functions (Eq. 4), have been drawn using the same value for v_0, namely $v_0 = 0.2$ (1/hr.) and for σ_0 the values shown. The construction of this second chart (Fig. 4) is an extremely simple matter. Only one single curve needs to be computed from points. For a given v_0 all curves corresponding to different values of σ_0 are obtained by multiplying the ordinates of the first curve by a constant factor. These curves must approach asymptotically the family of straight lines converging in the point $v_0/2 = 0.1$ (1/hr.), $\sigma = 0$ (for $v_0 = 0.2$ (1/hr.) and in addition all curves approach asymptotically also the abscissa axis.

We shall now explain how this chart has to be used. To make use of it for a determination of the two unknown constants v_0 and σ_0, it is necessary only to plot the points v_1, σ_1; v_2, σ_2, ... representing two or more creep tests, on a sheet of transparent paper with which the chart has been covered, using any desired stress scale but *the same* logarithmic creep rate scale as the one appearing on the chart. It is recommended to trace on the transparent sheet only one horizontal line, which is kept coincident with the v axis of the chart. *After all the points have been marked on the loose sheet, it is required to move it horizontally until the test points fall nearest to one of the curves of the chart.* If only *two* points have been used from creep observations, the sheet has to be moved until these two points lay on one of the curves or appear equally placed between two consecutive lines. The desired value of v_0 is read on the movable horizontal scale just above the "target," which was indicated on the chart, and the value of the constant σ_0 is read on the line passing through the given points (or interpolated from the two lines nearest to these points).

If the stress scale on the movable sheet has been taken such that one stress unit on the chart equals 100 or 1000 psi. or 100 or 1000 kg. per sq. cm., it is necessary only to multiply the value of σ_0 shown on the chart (Fig. 4) by the same factor and use the same units. This factor may have any desired value so long as σ and σ_0 are multiplied by the same number. This made it possible to design a single chart which can be used to represent any stress-creep rate relations without conversion factors.

It is obvious that the constants just obtained cannot be applied for an extrapolation to low values of creep rates if the logarithmic law (Eq. 2) is used or the equivalent of it, namely, the converging straight lines which were shown on the first chart (Fig. 3) since they would lead to a finite creep rate when the stress drops to zero. For the extrapolation to low values needed in machine design, use has to be made of Eq. 4

$$v = v_0 \sinh \frac{\sigma}{\sigma_0}$$

in which v_0 and σ_0 are the two constants just determined.[13]

Those who prefer to use a curve for the extrapolation (instead of Eq. 4 with the values of v_0 and σ_0) *may retrace the curve which fitted best the observation points with pencil on the transparent piece of paper and make further use of it directly.*

For large creep rates (which have recently become of practical interest under certain conditions when short life of the machine parts is expected) the first chart can be used expressing the logarithmic speed law under the same rules assuming that otherwise conditions are favorable for application of this law.

In applying both charts attention should be paid to the scales for stress σ and creep rate v. These latter may be referred to seconds, minutes, hours, etc., without changing the figures printed below the chart. Stress may be expressed in pounds per square inch or kilograms per square centimeter and be multiplied by any desired factor, such as 0.1, 10, or 100 without change in curve form *provided that the printed figures expressing the constants σ_0 are multiplied by the same numerical factor.* This permits use of the same chart for plotting of data from various sources over very different ranges of the rates or stresses from weak to strong materials. The range of creep rates covered is considered ample for any practical problem. If the range requires extension, lower values are easily handled

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[13] Computation of points on the curve represented by Eq. 4 is facilitated by the tables of the hyperbolic sine function for example by: Keiichi Hayashi, "Fünfstellige Tafeln der Kreis- und Hyperbelfunktionen," Walter de Gruyter and Co., (Berlin) (1938), or "Tables of Circular and Hyperbolic Sines and Cosines for Radian Arguments," Works Progress Administration, National Bureau of Standards (1939).

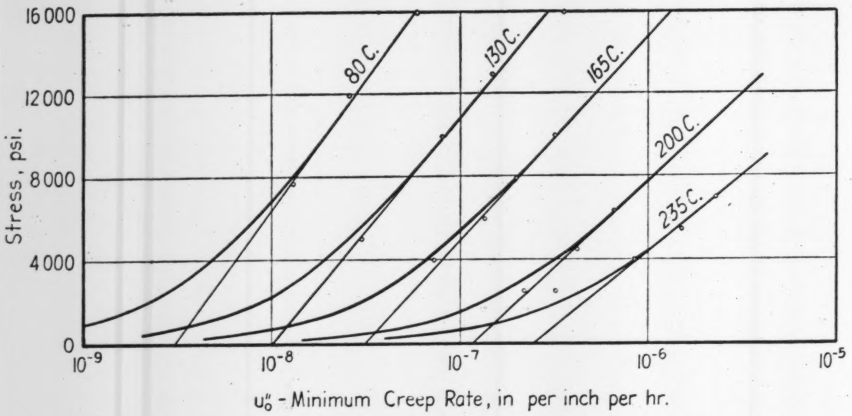

FIG. 5.—Stress-Creep Rate Relations for Oxygen-Free Copper (E. A. Davis).

since the hyperbolic sine is equal to the angle in radians to one part in the fifth decimal place for values below 0.031. In the field of high creep rates, uniformly spaced linear curves may be used.

Comparison of Eqs. 1 and 5 shows that in the semilogarithmic plot the choice between the hyperbolic sine and its asymptote depends upon the error introduced by the simpler assumption. This error may be evaluated by transposing Eqs. 1 and 4 as follows:

$$\sigma_1 = \frac{\sigma_0[\log v - \log v_0 + \log 2]}{\log_{10} e} \quad (12)$$

$$\sigma_2 = \sigma_0 \sinh^{-1}\left(\frac{v}{v_0}\right) \ldots\ldots (13)$$

The percentage difference in terms of stress at any value of creep rate is

$$\frac{\sigma_2 - \sigma_1}{\sigma_2}100 = \frac{\sinh^{-1}\left(\frac{v}{v_0}\right) - \frac{\log v - \log v_0 + \log 2}{\log_{10} e}}{\sinh^{-1}\left(\frac{v}{v_0}\right)} \times 100 \ldots\ldots (14)$$

This difference at any value of creep rate, v, is independent of the stress and the constant σ_0. It may be represented, therefore, on either chart (Fig. 3 or 4) by vertical straight lines which represent boundaries between areas on the charts in which the replacement of the hyperbolic sine by its asymptote results in stress differences between specified limits. Four lines are shown on Figs. 3 and 4 representing boundaries for differences of 0.1, 0.5, 1.0, and 5.0 per cent. In each case, the use of the logarithmic speed law in the area to the right of the vertical line introduces a stress difference less than the specified value.[14]

From the fact that the hyperbolic sine function is a monotonously increasing function of its argument, it follows that it is *not always possible* to draw a function of this kind through *two arbitrarily given points.* Evidently these two points must satisfy the requirement that the ordinate of the point having the smaller abscissa must be smaller than the ordinate of the point having the larger abscissa. If for example

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[14] An indication that test observations follow the logarithmic speed law over the range studied does not imply that the straight line can be extrapolated to lower creep rates. Whether the difference between the hyperbolic sine and its asymptote is large or small, the constants v_0 and σ_0 must be used in Eqs. 4 or 5 and not in Eqs. 1 and 2 to compute stresses for low values of creep rate.

$v_1 < v_2$ but $\sigma_1 > \sigma_2$, a solution for σ_0 and v_0 does not exist. Two or more than two observations among which this condition would exist are incompatible with Eq. 4.[15]

It is in order to quote a few test results which can serve to illustrate that various creep observations seem to follow the trends explained in the preceding discussion. In Fig. 5 are reproduced the results of creep tests for oxygen-free copper run at 80 C., 130 C., 165 C., 200 C., and 235 C. by Davis.[16] A series of repeated relaxation tests of a 0.35 per cent carbon steel (A.S.T.M.-A.S.M.E. Joint Research Committee, designation K-20) at the comparatively high temperature of 1100 F. were made by G. H. Heiser in our Laboratories in 1939. The unpublished test results are shown in Fig. 6. These curves were found to agree with the general shape of relaxation curves as predicted by the theory based on the hyperbolic sine creep law.[17]

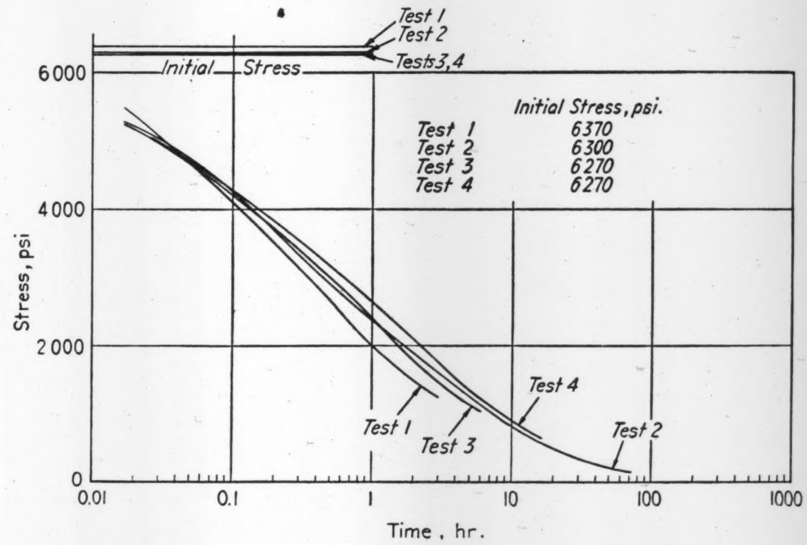

FIG. 6.—Repeated Relaxation Tests of 0.35 per cent Carbon Steel, K-20, at 1100 F. (G. H. Heiser).

In a third example a few creep tests of lead are quoted. These were described among a large group of creep tests in an investigation by Moore, Betty, and Dollins.[18] These authors have published a number of creep-rate stress diagrams for lead and lead alloys in which they represented their test results in double logarithmic plots. Among the many such plots just a few are fairly good straight lines, but the majority of these curves show a downward curvature in the same manner as the curve in our Fig. 2. They write: "It would seem, then, that under low stresses in lead and lead alloys the Bailey equation does not hold" (referring to the power function law). Also: "This suggests that the creep of lead may start out with stress-flow relations much like those of a truly viscous liquid and that as stress increases these relations change and the rate of creep increases faster than the increase of stress." In Fig. 7 three series of these creep tests were replotted in the manner described in this paper and the constants v_0 and σ_0 were estimated from the chart, Fig. 4, their values are given in Fig. 7.

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[15] The same restriction applies to the power function. If $v_1 < v_2$, but $\sigma_1 > \sigma_2$ the exponent m in Eq. 3 could not remain positive and extrapolation to lower creep rates would yield the unreasonable fact that they would result from higher stresses.

[16] E. A. Davis, "Creep and Relaxation of Oxygen-Free Copper," *Journal of Applied Mechanics*, Vol. 10, No. 2, June, 1943, p. A-101.

[17] A. Nadai, "The Creep of Metals Under Various Stress Conditions," Applied Mechanics, Theodore von Karman Anniversary Volume, Fig. 4, p. 247 (1941).

[18] H. F. Moore, B. B. Betty, and C. W. Dollins, "Investigation of Creep and Fracture of Lead and Lead Alloys for Cable Sheathing," *Bulletin No. 306*, University of Illinois, August, 1938.

Herbert F. Moore, and N. J. Alleman, "The Creep of Lead and Lead Alloys used for Cable Sheathing," *Bulletin No. 243*, University of Illinois, 1932.

Herbert F. Moore, B. B. Betty, and C. W. Dollins, "The Creep and Fracture of Lead and Lead Alloys," *Bulletin No. 272*, University of Illinois, February, 1935.

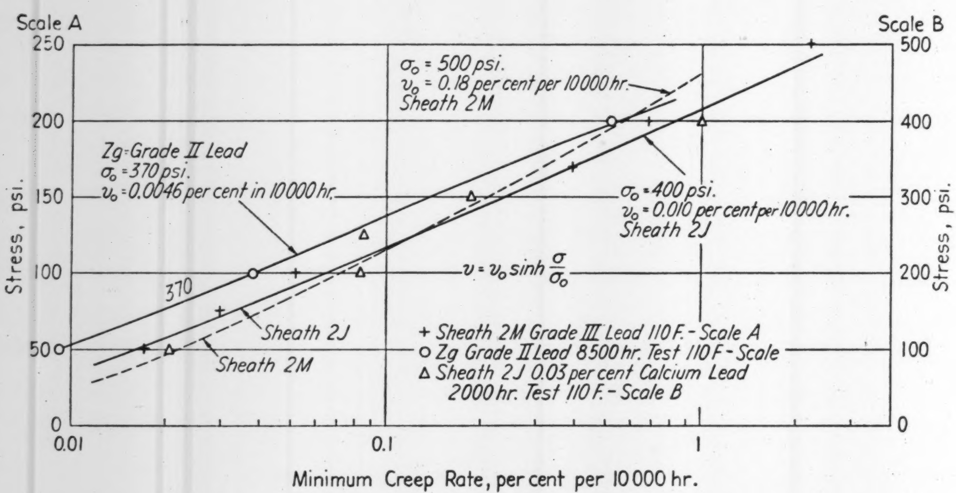

FIG. 7.—Creep Tests of Lead and Lead Alloys at 110 F. (H. F. Moore, B. B. Betty, and C. W. Dollins).

A last example refers to a group of creep tests reported in the Creep Data Book of 1938[19] for a wrought 18 per cent chromium, 8 per cent nickel steel tested at 1500 F. This group of creep tests was discussed also by one of the authors recently.[4] From this book the following values were taken:

Stress, psi.	Creep Rate $\left(\frac{1}{1000\text{ hr.}}\right)$
1500	0.00012
2000	0.00025
3000	0.00128
4000	0.00644

After plotting these four points on transparent paper laid above the chart Fig. 4 (Plate IV) and after moving the sheet of paper by the required amount for the constants v_0 and σ_0 the following values were obtained:

$$v_0 = 0.0020\left(\frac{1}{\text{hr.}}\right), \qquad \sigma_0 = 620 \text{ psi.}$$

Hope is expressed that the chart (Fig. 4) is sufficiently accurate for many practical applications. Further experience in the use of it may reveal limitations which restrict its application to practical problems. This would for example be expected for unstable alloys whose aging or precipitation hardening during the testing or service time may produce sets of creep-time curves of unusual shapes. Application of the chart is restricted to tests made at one and the same temperature. Even slight variations from this requirement may result in appreciable departures from any functional relation valid for constant-temperature tests. This restriction applies to any attempts to establish functional relations between stress and creep rate. Factors of this kind rule against the use of short duration creep tests to predict behavior of such materials during a long service time under the present state of our knowledge of the creep characteristics of such alloys.

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[19] "Compilation of Available High-Temperature Creep Characteristics of Metals and Alloys," published jointly by The American Society of Mechanical Engineers and American Society for Testing Materials (1938).

CONCLUSION

In conclusion this paper attempted to show that the hyperbolic sine function is more suitable to express the dependence of the rate of creep on the stress over a wide range of rates than the power functions which have almost exclusively been used for this purpose to date. The former function leads to safer estimates of the stresses and its ease of application has been facilitated through the chart, by means of which it becomes a relatively simple matter to survey the field of existing data and to express the behavior of alloys at high temperatures in easily comparable form.

DISCUSSION

Mr. Howard C. Cross[1] (*presented in written form*).—The authors have done a very complete job in showing us the results of their years of study of this creep problem. There can be little doubt that some of the available creep data more nearly follow the hyperbolic sine function than the power function.

The establishment of this relationship for a certain material in a known condition is ample justification for extrapolation in a like manner from tests of short duration for material of similar composition and treatment.

Structural stability of a material is a factor not taken into consideration in any mathematical extrapolation of creep rates. It is unwise to attempt extrapolation where stability is not certain. For this reason I hope neither this nor any mathematical analysis will influence people to extrapolate rather than run actual tests, but rather to determine directly the effect of stability both during and after the tests, particularly in the case of comparatively unknown new materials.

Mr. J. J. Kanter.[2]—The authors have made a very interesting analysis of the constant creep rate relationship to stress for metals and undoubtedly have described a method for expressing this relationship more satisfactorily, under some circumstances, than has been frequently used. Although the hyperbolic sine relationship seems to correlate much data with an amazing degree of precision, apparently, it cannot be used to extrapolate high stress data toward low rates unless experiment has established that this relationship is valid for the particular conditions of creep involved. On the strength of statements made by Mr. McVetty, it seems that something like half of the data surveyed is satisfactorily correlated by this analysis. While for many problems, the tendency which the analysis has to extrapolate stresses on the conservatively low side, there are other problems for which the importance of creep research lies in determining the largest stress feasible.

The impression is gained from what the authors have shown that the hyperbolic sine analysis gives a good correlation for data taken at temperatures high enough definitely to promote recrystallization of slip bands. Under such circumstances the analysis may be reliable. Where the analysis seems to break down is for the lower ranges of temperature at which creep is studied, where work hardening influences still predominate and tend to enhance the strength of the material. In this range of temperature, it appears an extrapolation of short-time points, particularly those corresponding to high deformation rates, by the hyperbolic sine method might lead to a seriously low estimate of the potentialities of materials needed for high-duty parts important to the war effort.

Whether the hyperbolic sine relationship has more than an empirical significance, useful in fitting a curve to some data, is not entirely clear. Constant creep rate has been attributed to an equilibrium between two principal effects, one which might be called strain hardening or slip band formation, the other annealing or recrystallization. Superimposed upon these effects may be other factors such as precipitation of additional phases from solid solution, spheroidizations and other structural changes. These effects may all influence the load-carrying ability of the metal. Presumably more than one constant would be needed to express the contribution of each of these important factors to the constant creep rate - stress relationship. To express the net effect, dependably, it is to be expected that more than the two constants of the hyperbolic sine relationship are needed. Therefore, whether this relationship reflects something fundamental to the mechanism of creep or is merely a fortuitous empirical device for fitting an established curve is a matter for deliberation.

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[1] Metallurgist, Battelle Memorial Institute, Columbus, Ohio.

[2] Materials Research Engineer, Research and Development Laboratories, Crane Co., Chicago, Ill.

MESSRS. A. NADAI[3] AND P. G. MCVETTY[3] (*authors' closure, by letter*).—Mr. Cross has mentioned the importance of structural stability. This factor must always be considered in any analysis of creep test data. In general, heat-resisting alloys may be divided into two broad classes. In one, the main requirement is high strength during a relatively short service life and stability is of secondary importance. Many of the alloys recent developed are of this type. In the other class, satisfactory service over a long period of time is the primary requirement and strength may be sacrificed, if necessary, to ensure structural stability. These factors are considered in the preparation of the materials, the formulation of suitable testing programs and in the analysis of the test data. In the class of materials first mentioned, the choice of working stresses can be based on tests having a duration equal to the expected service life. In this way extrapolation is eliminated and lack of structural stability is less important than assurance that the material tested is representative of that actually used. When a long service life is expected, any appreciable change in the strength of the material while in service would defeat the purpose of preliminary tests.

If a metal shows the signs of metallurgical structural instability or if phase changes occur within the total life of a construction piece and the creep tests have only covered a relatively small fraction of this time, the authors agree with Mr. Cross that conditions need further considerations before the test results can or should be extrapolated in the manner proposed in the paper. It would seem premature to include such cases of creep in an analysis until the new variables have been discovered which should serve to define *mechanically* what is meant by the term "structural instability" which has been used by metallurgists only in a metallurgical sense.

Mr. Kanter objects to the use of the hyperbolic sine function as a means for extrapolating creep tests toward the lower stress region but fails to provide a satisfactory or a better theoretical or practical substitute. Although admittedly the available test data cover a limited stress range, we have considerable experimental evidence to support the logarithmic speed law $\sigma = \sigma_0 \log_e \left(\frac{v}{v_0}\right)$ at the higher rates of creep. As the authors have pointed out in the paper, sound *mechanical* reasons require that the stress σ in this speed law should be expressible by an odd function of the rate of flow v. This point should be emphasized. It is accepted that creep in tension is similar to creep in compression, both being essentially shear rate phenomena not dependent on the mean normal stress. This statement is further verified through recent creep tests in pure bending. It is very probable that therefore $v = 0$ when $\sigma = 0$ and that at very small stresses or rates creep must follow the laws of pure viscous flow. These concepts help to visualize the hyperbolic sine function as the simplest of functions which satisfies these requirements and which represents a curve passing through the origin $v = 0, \sigma = 0$ with a finite slope. Mr. Kanter himself favored this latter assumption in one of his former papers but he nevertheless prefers in the lower range of temperatures to use now some unidentified function or functions for this extrapolation of the curve $\sigma = f(v)$ toward the smaller values of v instead of the function proposed by Eq. 4 of our paper because, as he expresses himself, the former ones should offer more potentialities for the use of high duty materials which are so important to the war effort. He has, however, not offered material to support his contention that the actual stresses are higher than the ones predicted by the hyperbolic sine function in this range of the rates of flow.

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[3] Consulting Mechanical Engineer, and Mechanical Engineer, respectively, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

Mr. Kanter sees a weakness in the hyperbolic sine function because it contains only two material constants. Our analysis of tests on a wide variety of materials has covered a fairly broad temperature range and we do not find it necessary to use a different method of analysis at the lower temperatures—*provided straight-line portions appear distinctly in the creep-time curves.* At very low temperatures in the pure strain-hardening range when a minimum creep rate is not clearly observable, there would be no point in using an equation like our Eq. 4 in which one of the variables refers to the minimum rate. The same is true if phase changes should occur during times which are small fractions of the time of testing or of service. Under these circumstances it seemed to us preferable to use a function satisfying the above mentioned general mechanical requirements and having the advantage of being based on two material constants instead of an indefinite number of such constants.

Mr. Kanter questions also whether this function reflects something "fundamental" to the mechanism of creep and whether it has more than an "empirical" significance for extrapolating creep data. The suggestion to extrapolate a curve rather than a straight line representing the test points (in either kind of a plot, double or semi-logarithmic) has been emphasized by Mr. Kanter in one of his recent publications. Drawing of such curves certainly must be called an "empirical" process of extrapolation. The authors believe that they could go one step farther in this respect. To their knowledge physical considerations seldom support power function laws having irrational exponents. They rejected these functions for this extrapolation for several reasons, although they believe that for another theoretical purpose they are still most valuable tools—namely, when dealing with creep under combined stresses, in which latter cases they have been found very useful for establishing exact relations for the stresses. But considerations of the above-mentioned nature lead them to believe, that the hyperbolic sine function for expressing the creep speed laws has been actually based successfully, also on theoretical grounds. In this connection we have referred to the recent theoretical investigations explaining the speed dependency of the flow of solids (Prandtl's theory of solids, Eyring's reaction rate theory and others) through which this latter function has received additional support.

THE EFFECT OF OVERSTRESSING AND UNDERSTRESSING IN FATIGUE

By J. B. Kommers[1]

Synopsis

Overstressing tests were carried out on smooth and square-notched specimens of annealed ingot iron and S.A.E. 1030 steel, and on smooth specimens of cold-drawn ingot iron. Damage curves are drawn for these cases, showing how the percentage of damage varies with the number of cycles of overstress.

Understressing tests were carried out on smooth specimens of annealed ingot iron. It is shown how the endurance limit is increased with increase of the number of cycles of understress. It is also shown how understress plus subsequent "coaxing" may further increase the endurance limit.

While understressing plus coaxing may increase the endurance limit from 25 to 30 per cent, it is shown that such stressing may increase the endurance life at higher stresses as much as 23,000 per cent.

The question why a material that is subjected to repeated stresses above the endurance limit finally develops a crack and fails has puzzled engineers for many years. There must be changes occurring in the material during the progress of the test, otherwise there would be no failure. Inelastic action and permanent deformation, as usually thought of, are not necessarily criteria for fatigue failure. For example, the maximum stress in a direct-stress cycle that is not completely reversed may be above the tensile yield point, so that at the first application of the upper limit of stress there is a large permanent set. Even in this case, if the lower limit of stress is properly chosen, fatigue failure will not occur. On the other hand, under completely reversed stresses, there may be no recognizable phenomena which would make one suspect that failure will take place, and yet failure occurs.

One of the main purposes of the present tests has been to determine the changes that occur during the progress of a fatigue test, in an endeavor to add to the information available regarding the phenomena of fatigue failure. In Great Britain, Gough and Wood[2] have investigated the changes which proceed under fatigue action by precise X-ray methods. The method employed by the author has been to subject specimens to overstress for various numbers of cycles less than those required for failure, and then to determine the new endurance limits. By this method the changes that may be proceeding in the material are measured by the change in the endurance strength of the material.

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[1] Professor of Mechanics, College of Engineering, University of Wisconsin, Madison, Wis.

[2] H. J. Gough and W. A. Wood, "A New Attack upon the Problem of Fatigue of Metals, Using X-ray Methods of Precision," *Proceedings*, Royal Soc. (London), Vol. 154A, p. 510 (1936).

The understressing tests show the changes that occur during a fatigue test when the stress is below the endurance limit. In general, both overstressing and understressing tests show that the endurance limit is being changed by such stressing.

TABLE I.—CHEMICAL ANALYSIS OF MATERIALS.

Material	Carbon, per cent	Manganese, per cent	Silicon, per cent	Sulfur, per cent	Phosphorus, per cent
Ingot iron, annealed...	0.015	0.03	0.01	0.050	0.007
Ingot iron, cold-drawn.	0.04	0.02	0.03	0.032	0.005
S.A.E. 1030 steel.......	0.32	0.75	0.21	0.027	0.020

TABLE II.—TENSILE PROPERTIES AND HARDNESS OF MATERIALS.

Material	Tensile Strength, psi.	Yield Point, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Rockwell Hardness	Brinell Hardness
Ingot iron, annealed.......	44 300	22 700	47.0	75.1	B53	83
Ingot iron, cold-drawn ..	73 100	69 200	11.7	63.0	B87	142
S.A.E. 1030 steel.........	80 400	44 600	32.3	62.0	B80.5	135

TABLE III.—ENDURANCE LIMITS OF MATERIALS.

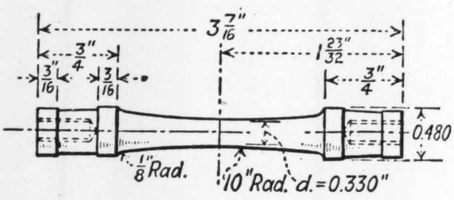
Material	Endurance Limit, Standard Specimen, psi.	Endurance, Limit, Square-Notched Specimen, psi.	Ratio of Endurance Limit to Tensile Strength	
			Standard Specimen	Notched Specimen
Ingot iron, annealed .	26 200	18 500	0.59	0.42
Ingot iron, cold-drawn..............	34 000	.....	0.57	...
S.A.E. 1030 steel......	37 200	23 800	0.46	0.30

Quite apart from the increase of endurance limit that understressing may confer on a material, is the increased number of cycles of stress that may be withstood successfully after understressing. Even when the increase of endurance limit is only of the order of 25 or 30 per cent, the increased "life" at higher stresses, as measured by the number of cycles, may be of the order of 23,000 per cent.

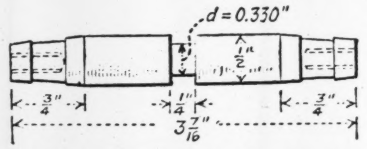
MATERIALS, TESTS, AND TEST SPECIMENS

Table I shows the chemical analysis of the materials used in the tests, and Table II shows some of the mechanical properties. Table III shows the endurance limits of the standard smooth and the square-notched specimens.

The annealed ingot iron was chosen for these tests because it is very homogeneous and has some unusual fatigue properties. The cold-drawn ingot iron was chosen as an example of cold-worked material, and the S.A.E. 1030 steel was chosen as an example of commonly used steel.



(a) Standard fatigue specimen.



(b) Notched fatigue specimen.

FIG. 1.—Fatigue Specimens.

The annealed ingot iron was furnished in the form of 1-in. diameter bars, the cold-drawn ingot iron as $\frac{1}{2}$-in. bars, and the S.A.E. 1030 steel as $\frac{3}{4}$-in. bars. In order to obtain greater uniformity in physical properties, the S.A.E. 1030 steel was heated to 825 C. and held for $\frac{1}{2}$ hr. at this temperature.

The tension test specimens of annealed

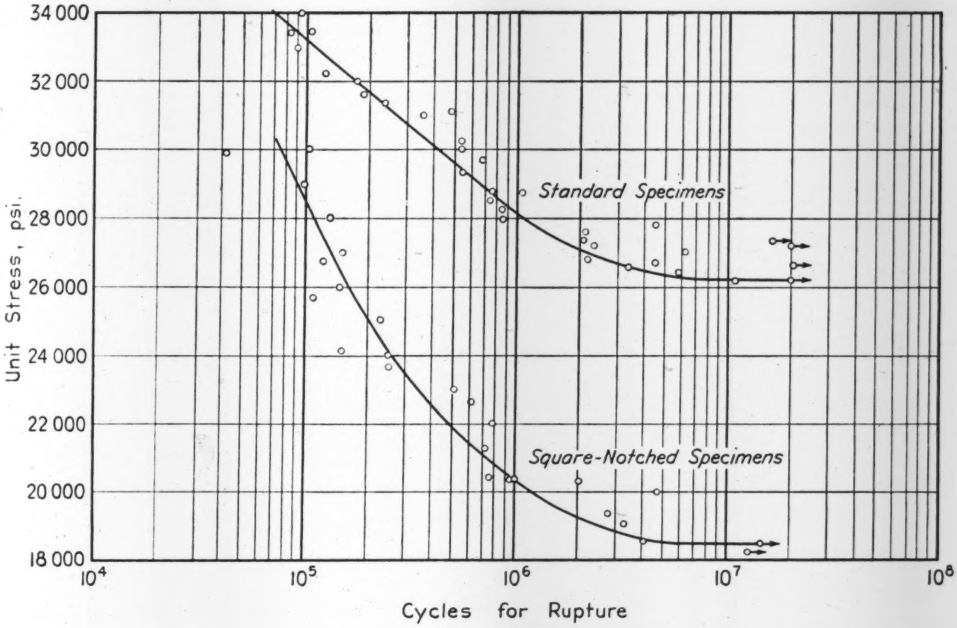


FIG. 2.—*S-N* Curves for Annealed Ingot Iron.

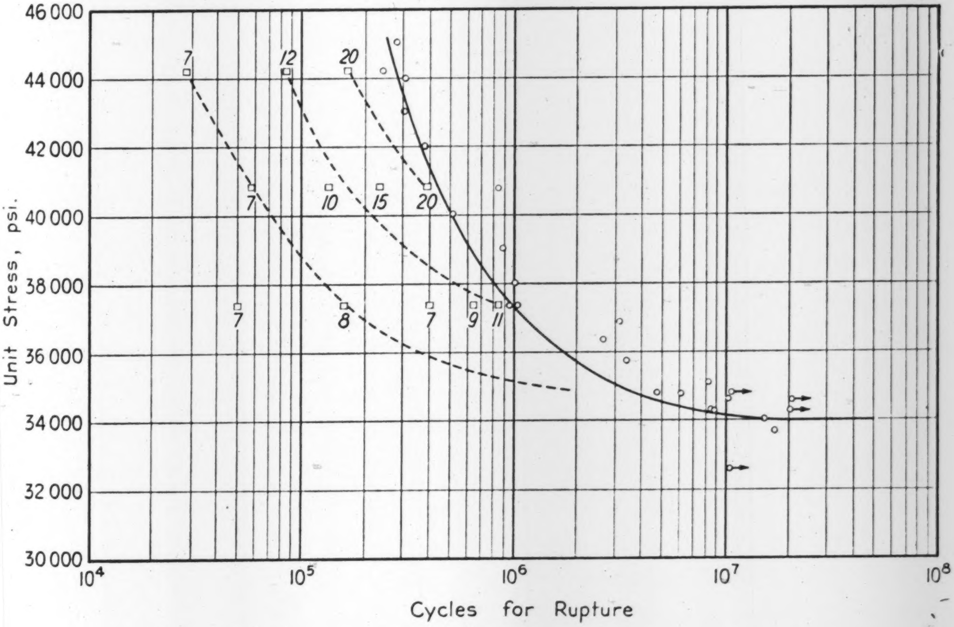


FIG. 3.—*S-N* Curve for Cold-Drawn Ingot Iron. Standard specimens.

ingot iron and S.A.E. 1030 steel were $\frac{1}{2}$ in. in diameter, and the cold-drawn ingot iron was $\frac{1}{4}$ in. in diameter.

Figure 1 shows the standard smooth and the square-notched fatigue specimens. The minimum diameter of the annealed ingot iron was about 0.33 in., while that of the cold-drawn ingot and the S.A.E. 1030 steel was about 0.30 in. The fatigue tests were made on R. R. Moore fatigue testing machines, which subject the rotating-beam specimens to completely reversed stresses in tension and compression. The speed of the fatigue machines was 1750 rpm.

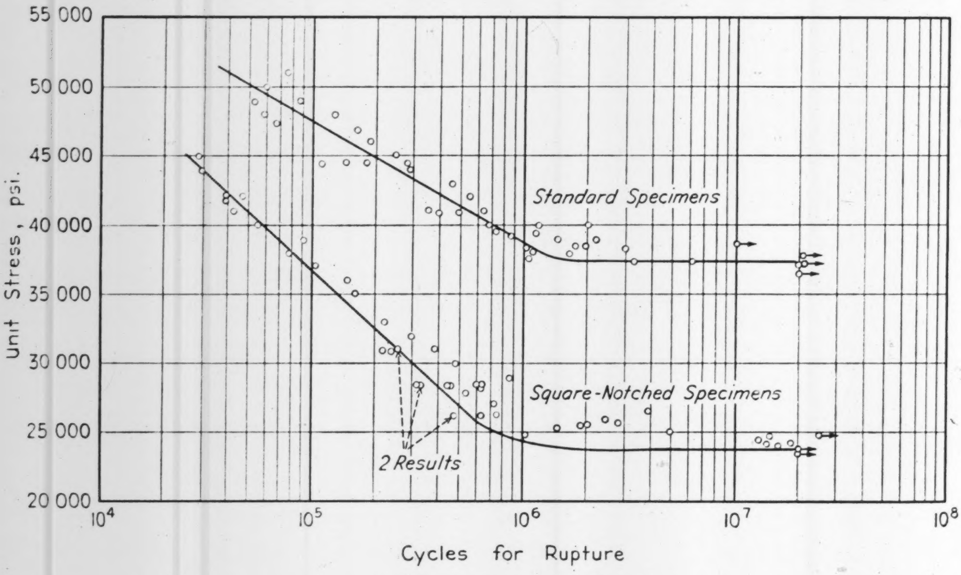


Fig. 4.—*S-N* Curves for S.A.E. 1030 Steel.

Figure 2 shows the *S-N* diagrams for smooth and square-notched specimens of annealed ingot iron. The solid curve in Fig. 3 shows the *S-N* diagram for cold-drawn ingot iron. Figure 4 shows the *S-N* diagrams for smooth and square-notched specimens of S.A.E. 1030 steel.

Overstressing Tests

In making overstressing tests, a series of specimens was subjected to an overstress of, say, 10 per cent above the endurance limit for a certain cycle ratio. The term cycle ratio means the ratio of the number of cycles used with the overstress to the total number of cycles which would cause failure at this stress. After overstressing, the load on the specimen was reduced in order to determine the new endurance limit. The number of specimens required for this purpose depended on the correctness of an estimate of the probable new value of the endurance limit. The stresses used were calculated as a percentage of the original endurance limit, and the new endurance limit was practically always based upon a pair of results which showed that specimen *A* did not fail, but that specimen *B*, with a stress 1 per cent higher than that used for specimen *A*, did fail. For the specimens that did not fail a total run of 10 million cycles was usually used, but sometimes 20 million cycles was also used. Damage due to overstress was expressed by the percentage that the new endurance limit was below the virgin endurance limit.

The upper part of Fig. 5 shows percentage of damage plotted against cycle ratio for standard smooth specimens of annealed ingot iron at 10 and 20 per cent overstress. For 10 per cent overstress, the value of 28,800 psi., shown on the graph, represents the stress used in the overstress tests, and the 720,000 cycles represents the average cycles for failure at 28,800 psi., as determined from the *S-N* diagram for this material.

The lower part of Fig. 5 shows damage curves for square-notched specimens at 10, 20, and 30 per cent overstress. The attempt was made to stress smooth specimens at 30 per cent overstress, but this stress was so far above the yield point that the specimens failed by bending.

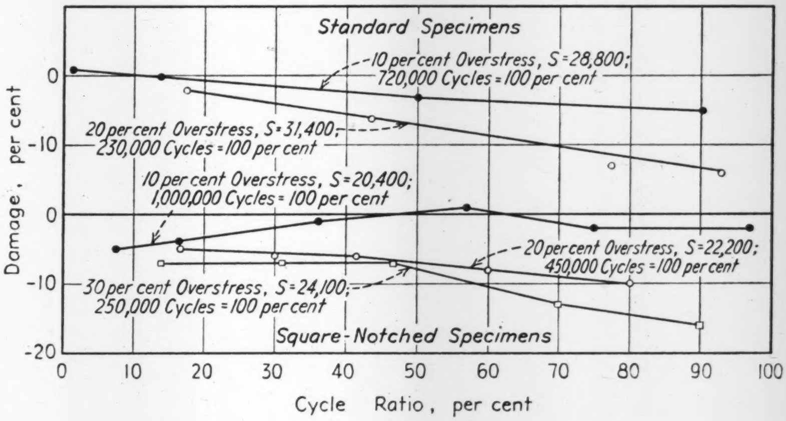


FIG. 5.—Damage Curves for Annealed Ingot Iron.

For the smooth specimens in Fig. 5, straight lines seem to fit the experimental points. There is some evidence that the endurance limit is slightly increased at very small values of cycle ratio. These specimens are not very susceptible to damage due to overstress, because even at 20 per cent overstress the damage is less than 15 per cent at cycle ratios larger than 90 per cent.

Annealed ingot iron has a curious property under fatigue which the author has not found in any other ferrous metal. The yield point in tension is 22,700 psi. and the endurance limit under reversed bending stresses is 26,200 psi., or 15.4 per cent above the yield point. It has often been pointed out that there is no correlation between the endurance limit and the elastic properties as found in a static test. When ingot iron is subjected in fatigue to the endurance limit stress, there must be inelastic action because the stress is above the yield point. However, as the test proceeds, the material seems to be able to recover, so that it finally becomes perfectly elastic to a value well above the original yield point.

In Fig. 5, at 10 per cent overstress for the square-notched specimens, the damage curve exhibits a curious recovery of endurance limit with increased cycle ratio. The damage is about 5 per cent at small cycle ratios, after which there is progressive recovery, until at a cycle ratio of 58 per cent the endurance limit shows an increase of 1 per cent. Thereafter there is again some decrease, which remains small, however, even up to a cycle ratio of 97 per cent. This is the only definite case of recovery of a damage curve that the author has found in any overstressing tests.

For 30 per cent overstress on square-notched specimens, the damage curve shows no actual recovery, but the tendency toward recovery seems to be indicated by the fact that the curve remains horizontal up to a cycle ratio of about 47 per cent.

Figure 6 shows damage curves plotted to a base of actual number of cycles instead of cycle ratios. The curves show the relatively rapid increase of damage with increase in number of cycles at the higher percentages of overstress.

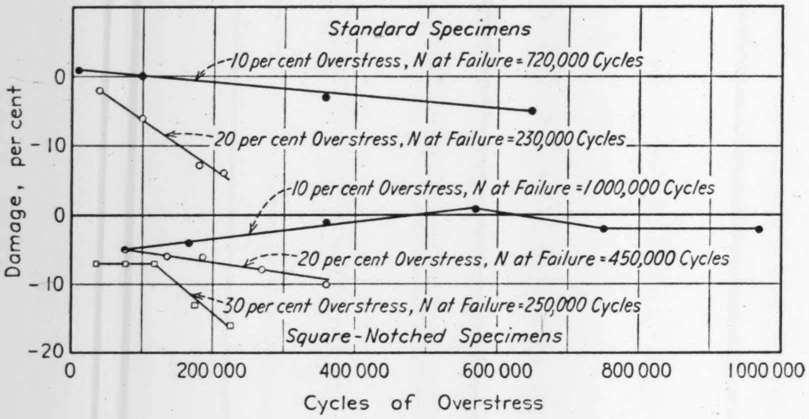


FIG. 6.—Damage Curves for Annealed Ingot Iron.

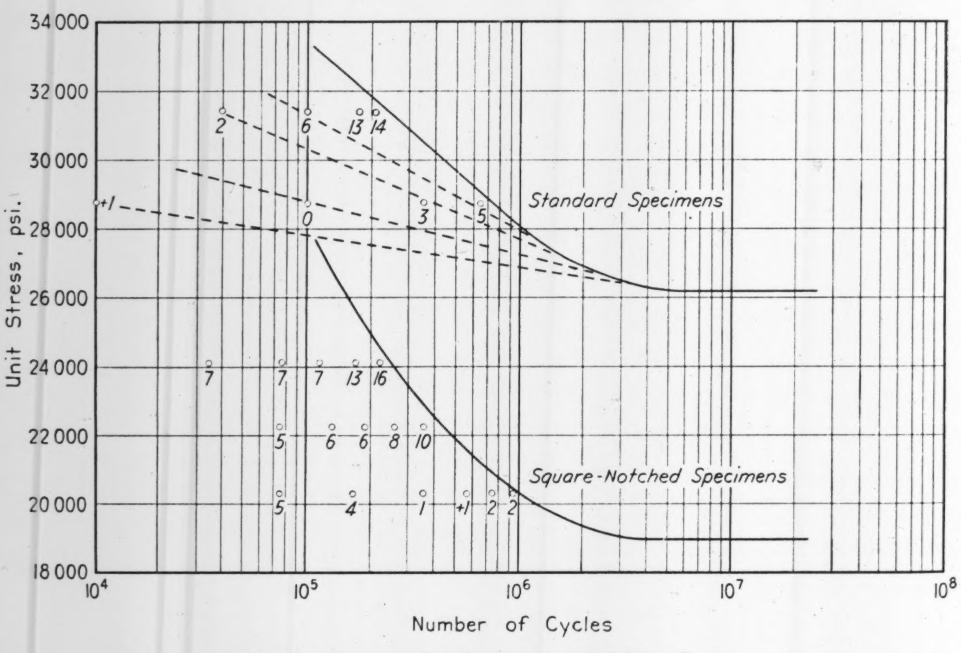


FIG. 7.—Damage Results for Annealed Ingot Iron.

In Fig. 7 the solid lines represent the

ordinary S-N diagrams for standard and square-notched specimens of annealed ingot iron. For the standard specimens the two horizontal rows of points represent the results obtained at 10 and 20 per cent overstress. The numbers adjacent to the plotted points represent the percentage of damage obtained at the various numbers of cycles. An attempt has been made to draw dashed lines which might correspond approximately to an increase of 1 per cent in endurance limit, and decreases of 0, 2 to 3, and 5 to 6 per cent.

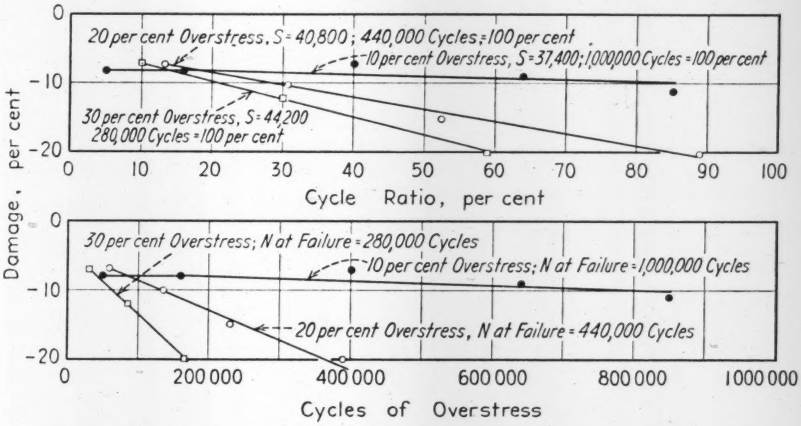


FIG. 8.—Damage Curves for Cold-Drawn Ingot Iron. Standard specimens.

In the lower graph of Fig. 7 the horizontal rows of points represent damage results obtained on square-notched specimens at 10, 20, and 30 per cent overstress. The numbers adjacent to the plotted points represent the percentage of damage found at the given stress and number of cycles. Because the damage curves in Fig. 6 did not show continually increasing damage with increase in number of cycles, it is evident that it is not possible to draw simple damage lines as was done by the dashed lines in the upper graph of Fig. 7. This is especially true for the damage results obtained at 10 per cent overstress.

Figure 8 shows damage curves for standard specimens of cold-drawn ingot iron. In the upper part of the figure, cycle ratio is used as a base, and in the lower part the actual number of cycles is used. For these results straight lines seem to fit the points fairly well, and none of the points fall above the line of zero damage. For this material there is about 7 per cent damage even for small numbers of cycles, and the damage increases with increasing numbers of cycles. Comparing Figs. 6 and 8, it is evident that cold-drawn ingot iron is much more sensitive to damage from overstress than is annealed ingot iron.

In Fig. 3 the dashed lines represent damage lines for standard specimens of cold-drawn ingot iron of 7 to 8 per cent, 11 to 12 per cent, and 20 per cent damage, respectively. Figure 8 shows that this material was fairly sensitive to overstress, but it is clear from Fig. 3 that when only 20 per cent of damage has been done at 20 and 30 per cent overstress, the corresponding number of cycles is quite close to the average S-N curve. These results indicate, therefore, that damage must increase very rapidly during the last portion of a fatigue test to failure.

The damage curves for standard specimens of S.A.E. 1030 steel are shown in Fig. 9. There is no evidence of recovery, and none of the points falls above the line for zero damage. The arrow on the last point plotted for 30 per cent overstress means that the damage was somewhat greater than that indicated.

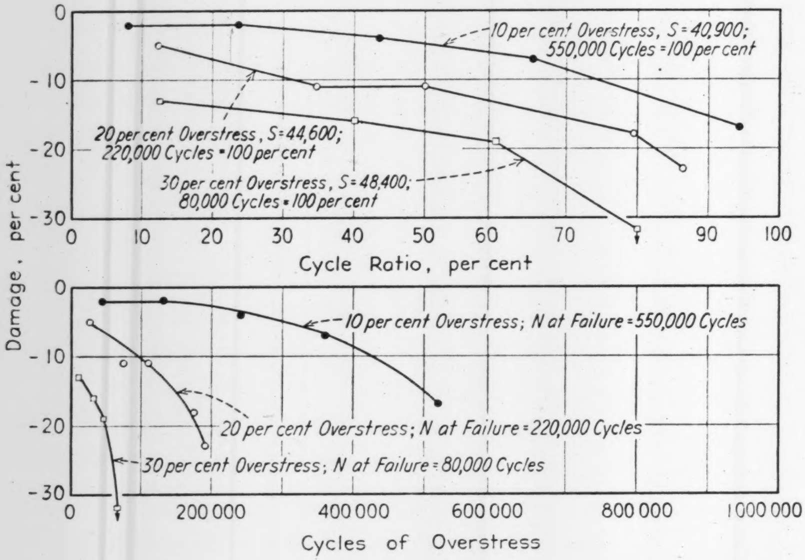


Fig. 9.—Damage Curves for S.A.E. 1030 Steel. Standard specimens.

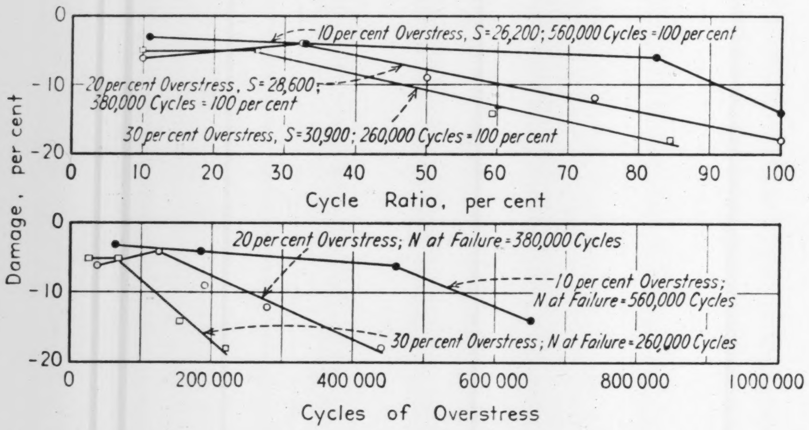


Fig. 10.—Damage Curves for S.A.E. 1030 Steel. Square-notched specimens.

The lower curves in Fig. 9 show what is probably characteristic when an overstressed specimen approaches failure. For instance, for the 20 per cent overstress curve, as the number of cycles approaches 220,000—the average number required to cause failure at this stress—the damage curves tend to become asymptotic to a vertical line. After 190,000 cycles of overstress the then existing endurance limit is only 77 per cent of the virgin endurance limit.

This means that the stress of 44,600 psi. being applied at this stage of a test represents an actual overstress of 56 per cent of the then existing endurance limit. With such a large increase of actual overstress and decrease of actual endurance limit, it is clear that only a comparatively few more cycles are necessary to produce failure.

Figure 10 shows damage curves for square-notched specimens of S.A.E. 1030 steel. None of the points falls above the line of zero damage. For the 20 per cent overstress curve there is some evidence of recovery at a cycle ratio of about 33 per cent. For the 10 and 20 per cent overstress curves, points are shown at a cycle ratio of 100 per cent. This means that a number of specimens were run to a greater number of cycles than the average cycles for failure, which were 560,000 and 380,000 cycles, respectively, at 10 and 20 per cent overstress.

From the above overstress results, it may be concluded that a material finally fails at a stress above the endurance limit because the endurance limit is being changed while the test proceeds. For some materials there may be actual recovery of endurance during part of the test, as shown by the 10 per cent overstress curve for square-notched specimens in Fig. 6. For other materials the endurance limit decreases gradually throughout the test, with a more rapid decrease before failure, as shown by the curves in Fig. 9. For still other materials the damage may remain fairly constant for most of the test, with the probability that the damage increases very rapidly just before failure. This is shown by the 10 per cent overstress curves in Fig. 8.

This last mentioned property of delayed damage would evidently be valuable for machine parts, such as rear axle gears in automobiles,[3] in which the working stress is deliberately chosen above the endurance limit. This is done because it is known that the life of the gears is greater than the life of the car.

The fact that for certain materials at small numbers of cycles the then existing endurance limit is above the ordinary endurance limit, that for square-notched specimens of ingot iron there was actual recovery of endurance limit, and that for some other materials there is delayed damage, seems to point to the conclusion that there is a destructive action and a strengthening action proceeding simul-

TABLE IV.—DAMAGE DUE TO OVERSTRESS.

NOTE.—Plus signs indicate the endurance limit increased; minus signs indicate the endurance limit decreased.

Material	Standard Specimen			Square-Notched Specimen		
	20 per cent Cycle Ratio	50 per cent Cycle Ratio	80 per cent Cycle Ratio	20 per cent Cycle Ratio	50 per cent Cycle Ratio	80 per cent Cycle Ratio
10 PER CENT OVERSTRESS						
0.27 per cent, carbon steel	−3	−9	−15	..	..	..
0.48 per cent carbon steel	−10	−20	−26	0	−3	−6
0.62 per cent carbon steel	−5	−10	−13	−8	−15	−20
Cast iron	−1	−2	−3	+5	+4	+3
Annealed ingot iron	−1	−3	−5	−3	0	−2
Cold-drawn ingot iron	−8	−9	−10	..	..	..
S.A.E. 1030 steel	−2	−5	−12	−3	−5	−6
20 PER CENT OVERSTRESS						
0.27 per cent carbon steel	−4	−15	−22	..	..	..
0.48 per cent carbon steel	−13	−20	−28	+3	−8	−11
0.62 per cent carbon steel	−11	−18	−25	−12	−19	−24
Cast iron	−8	−8	−9	+1	0	−1
Annealed ingot iron	−2	−7	−12	−5	−7	−10
Cold-drawn ingot iron	−8	−14	−19	..	..	..
S.A.E. 1030 steel	−7	−11	−18	−5	−8	−14
30 PER CENT OVERSTRESS						
0.27 per cent carbon steel	−7	−18	−30	..	..	..
0.48 per cent carbon steel	−20	−24	> −45	0	−6	−8
0.62 per cent carbon steel	−13	−20	−27	−12	−20	−24
Cast iron	−11	−14	−18	+5	+4	+3
Annealed ingot iron	..	..	..	−7	−8	−15
Cold-drawn ingot iron	−10	−17	..	..	..	..
S.A.E. 1030 steel	−14	−18	> −32	−5	−11	−18

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[3] J. O. Almen and A. L. Boegehold, "Rear Axle Gears: Factors Which Influence Their Life," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 99 (1935).

taneously. Evidently for stresses above the endurance limit the destructive action finally prevails and causes failure.

A surprising, and at the same time an encouraging, fact brought out by the overstress tests is that the materials are not damaged as much as might be expected by overstresses, and that they can withstand overstress for a very large proportion of the normal life without a really serious reduction of endurance limit. It should be remembered, however, that while this may be true for 10 per cent of overstress, it is not true for heavy overstress. Table IV summarizes these facts and includes results published by the Society in 1938.[4]

In Table IV, at 10 per cent overstress, it will be noted that the damage at 50 per cent of the normal life is in most cases not very serious, and there is no alarming increase of damage in going from 50 to 80 per cent of the normal life. However, at 30 per cent overstress and at 80 per cent of the normal life, the damage for standard specimens is considerable. Such results suggest that occasional overstressing, if the stress is not too high, may not seriously damage a material.

UNDERSTRESSING TESTS

In 1930 the author[5] reported understressing tests on cast iron that had tensile strength of 20,000 psi. After understressing at 9000 psi. (endurance limit 9300 psi.), it was found that the endurance limit could be increased 25 per cent at 15 million cycles. Beyond 15 million cycles, up to 40 million cycles, there was no further strengthening.

In the present experiments, similar tests were made on standard smooth specimens of annealed ingot iron having an endurance limit of 26,200 psi. A unit stress of 26,000 psi. was applied for various cycles up to 60 million. In these tests the higher stress applied after understressing was kept constant, and a maximum run of 20 million cycles was used for those specimens that did not fail. The results are shown in Table V.

Table V shows that the increase in endurance limit with increase of understressing cycles is not very regular. Between 30 and 40 million cycles there is a considerable jump in fatigue strength up to 19 per cent, and at 60 million cycles there is a further increase up to 23 per cent. These results indicate that annealed ingot iron, in contrast with cast iron, requires comparatively long runs of understressing to obtain substantial increases of fatigue strength.

TABLE V.—UNDERSTRESSING RESULTS.

Stress Used in Understressing, psi.	Number of Cycles of Understressing	Increase in Endurance Limit, per cent
26 000	5 000 000	6.1
26 000	10 000 000	6.1
26 000	20 000 000	6.9
26 000	30 000 000	8.0
26 000	40 000 000	19.0
26 000	60 000 000	23.0

Because of the lack of material, only a few tests were made at 80 million cycles of understress, and these seemed to indicate that there was no further increase of fatigue strength.

In determining the new endurance limit in the above tests, quite a number of specimens did not fail. These specimens were tested to failure by adding $\frac{1}{4}$, $\frac{1}{2}$, or 1 lb. per day until failure occurred. Each day's run involved an addition of about 2.5 million cycles. Furthermore, after it was found that 10 million cycles increased the endurance limit 6 per cent, a number of specimens were tested at 26,000 psi. for 10 million cycles, then a second run of 10 million cycles at 6 per cent overstress, and finally various "coaxing" increments were progressively added. Table VI shows the results obtained from these tests.

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[4] J. B. Kommers, "The Effect of Overstressing and Understressing in Fatigue," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 249 (1938).

[5] J. B. Kommers, "The Effect of Under-Stressing on Cast Iron and Open-Hearth Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 368 (1930).

Column 1 in Table VI shows the understress cycles at 26,000 psi. Column 2 shows the number of cycles of the next run at the higher stress shown in column 3. Column 4 shows approximately the increment of stress added thereafter per day, per two days, or per four days. One half per cent means ¼ lb. was added, 1 per cent means ½ lb. was added, and 2 per cent means 1 lb. was added. Column 5 shows the total number of cycles at failure, and column 6 shows the percentage of stress above the original endurance limit that was acting at failure.

TABLE VI.—RESULTS OBTAINED BY COAXING.

Test	Original Run, millions	Second Run, millions	Unit Stress in Second Run, Above Endurance Limit, per cent	Increment in Unit Stress in Succeeding Runs	Total Cycles at Failure, millions	Unit Stress at Failure, Above Endurance Limit, per cent
	1	2	3	4	5	6
No. 1	5.1	21.2	6.1	2% per day	37.5	16.8
No. 2	10.1	19.8	6.1	2% per day	39.9	14.9
No. 3	10.1	9.9	6.1	2% per 2 days	75.3	29.8
No. 4	10.0	10.2	6.1	2% per 4 days	120.3	27.5
No. 5	10.2	10.1	6.1	2% per 4 days	129.9	30.2
No. 6	10.3	10.2	6.1	Add 2% for 1 day Remove 1% for 1 day Add 3% for 1 day	79.6	29.0
No. 7	10.2	10.2	6.1	1% per day	92.0	36.3
No. 8	9.7	10.7	6.1	1% per 2 days	118.7	27.5
No. 9	10.3	10.2	6.1	½% per day	141.6	31.7
No. 10	20.5	20.6	6.9	2% per day	73.5	35.1
No. 11	30.3	20.5	8.0	2% per day	81.4	34.4
No. 12	40.0	21.4	17.9	2% per day	75.2	30.6
No. 13	41.2	17.9	8.0	2% per day	68.1	16.0
No. 14	41.0	20.4	9.2	2% per day	86.7	31.4
No. 15	40.7	21.3	9.9	2% per day	89.0	34.0
No. 16	40.9	20.0	19.1	1% per day	84.7	30.2
No. 17	40.6	20.3	11.8	1% per day	100.6	29.0
No. 18	41.0	20.0	11.1	1% per day	106.2	30.5
No. 19	40.3	20.9	14.1	½% per day	141.7	30.9
No. 20	61.5	20.4	14.1	2% per day	103.2	33.6
No. 21	61.6	20.0	8.8	2% per day	105.4	30.1
No. 22	60.9	20.3	22.1	2% per day	88.0	28.6
No. 23	60.6	20.6	17.9	1% per day	102.5	27.9
No. 24	60.6	20.7	17.9	1% per day	102.6	27.9
No. 25	60.4	20.5	19.8	1% per day	103.5	30.9
No. 26	61.2	21.2	13.0	1% per day	92.8	18.3
No. 27	60.3	20.4	22.9	1% per day	80.7	24.0

In Table VI it appears probable that tests Nos. 13 and 26 are lower for the stress at failure than might have been expected, judging from the other results. Tests Nos. 1 and 2 indicate that the "coaxing" increment of 1 lb. per day was probably too high for optimum strengthening, because tests Nos. 3, 4, and 5, which had increments of 1 lb. in 2 days and 1 lb. in 4 days, failed at a much higher stress than did tests Nos. 1 and 2. Apparently an increment of 1 lb. every 4 days produced no better results than 1 lb. every 2 days, even though the number of cycles in the first case was very much larger.

In test No. 6 the coaxing process was varied. After the first two runs of 10 million each, 1 lb. was added for one day. Thereafter, ½ lb. was taken off for a day, and 1½ lb. added the following day. This procedure gave the specimen a period of recovery of 2.5 million cycles at a lower stress every other day, and amounted to a 1 lb. increment every 2 days. This method apparently was no better than that used for test No. 3 which added 1 lb. every 2 days.

For tests Nos. 7 and 8, after the first two runs of 10 million cycles each, further coaxing was used at ½ lb. per day and ½ lb. per 2 days. There seemed to be no advantage in the second case. In test No. 9 coaxing was done at ¼ lb. per day, but showed no advantage over test No. 7.

In tests Nos. 10 and 11 the first two runs involved a total of 40 and 50 million cycles, respectively. After these longer initial runs, evidently coaxing with 1 lb. per day was not too large an increment, since excellent strengthening was obtained.

In tests Nos. 12 to 19, inclusive, the first run was about 40 million and the second about 20 million. It will be noted that the increment of stress added for the second run varied from 8 to 19 per cent, but the stress at failure was approximately the same in all cases, test No. 13 excepted. The coaxing with small increments was done at 1 lb., ½ lb., and ¼ lb. per day, with little difference in the stress at failure.

In tests Nos. 20 to 27, inclusive, the first run was about 60 million and the second about 20 million. The increment of stress added for the second run varied from 9 to 23 per cent. It seems probable that some of these increments were too high to produce optimum strengthening. For instance, in test No. 27, after coaxing began at ½ lb. per day, the specimen failed almost immediately after the first ½ lb. was added.

Table V shows that an initial understressing run of 5 or 10 million cycles increases the endurance limit about 6 per cent, while a run of 60 million cycles may increase the endurance limit as much as 23 per cent. This clearly indicates that long runs just below the endurance limit are more effective than short runs.

Table VI shows that after the first two runs coaxing will increase the endurance limit further. It is true that usually the runs used in coaxing were for only 2.5 million cycles, so that the actual endurance limit was not being obtained at these higher stresses. However, it appears that when the stress used in coaxing is not too large, the final stress at failure is not so greatly different no matter what coaxing method is used. This result would lead one to conclude that there is an upper limit beyond which strengthening in fatigue cannot be carried further.

The understressing tests on cast iron reported by the author in 1930[5] indicated that the optimum strengthening was obtained by using a stress just under the endurance limit. In this connection, it would be interesting to carry out a more elaborate series of tests than those reported in this paper. Test a series of specimens for 10 million cycles, say, just below the virgin endurance limit, and determine the new endurance limit. If this is, say, 6 per cent higher than before, test a second series for 10 million cycles just below the virgin endurance limit, then 10 million cycles at 5 per cent higher, or just below the second endurance limit, and determine a third endurance limit. Continue this proce-

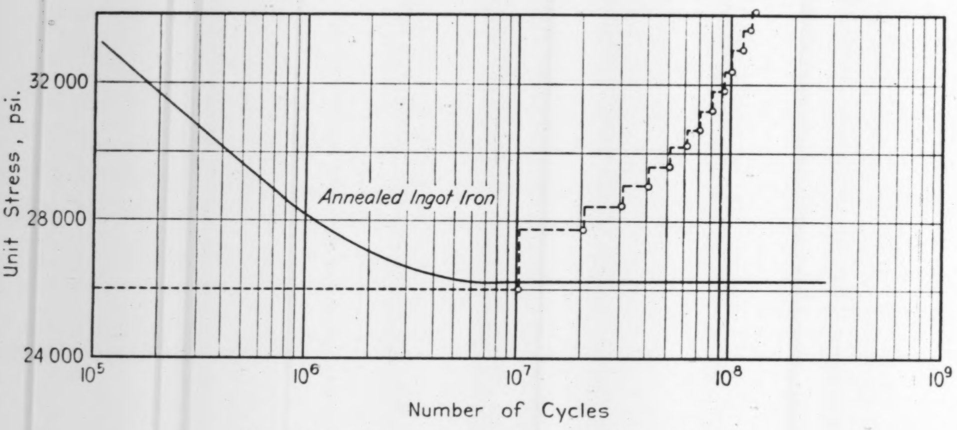

FIG. 11.—Results of Understressing and Coaxing.

dure so that the specimen is being tested in each run just below the endurance limit established by the previous steps of 10 million cycles each. In this manner it would be possible to determine what the upper limit is that can be obtained for strengthening by understressing operations.

While the effect of understressing and subsequent coaxing is quite marked in the increase obtained for the endurance limit, and in the increase of the stress which can be applied for an appreciable number of cycles, the effect is even more marked when examined from the standpoint of the greater life or number of cycles that the material can endure at the higher stresses. Figure 11 shows this very plainly. The smooth curve represents the *S-N* diagram obtained on standard smooth specimens of annealed ingot iron. The stepped curve shows the operations that were performed in the case of test No. 6 taken from Table VI. Each run consisted of about 10 million cycles, and after each run the load was increased by 1 lb.

The next to the last run was for a full 10 million cycles at a unit stress of 33,600 psi., or 28 per cent above the original endurance limit. The normal life of a specimen at this unit stress was about 85,000 cycles. Therefore, a run of 10 million cycles represents an increased life that is 118 times the normal life, or an increase of 11,700 per cent. This, however, does not represent the full increase in the endurance life at this unit stress. Since the specimen ran another 7 million cycles at a higher stress, it is probable that it would have run at least another 10 million cycles at 33,600 psi. This would be 235 times the normal life, or an increased life of 23,400 per cent.

Even at the highest stress applied, which was 34,100 psi., the specimen ran over 7 million cycles before failure. The normal life at this unit stress was about 65,000 cycles. The run of 7 million cycles represents an increase in life of 108 times, or 10,700 per cent.

Diagrams similar to Fig. 11 could be drawn for each of the tests listed in Table VI. However, in most of these tests the runs were made for only 2.5 million cycles for each increment used in coaxing. These tests, therefore, established the endurance limit for 2.5 million cycles, but not for indefinite endurance.

Table V shows that understressing may increase the endurance limit of annealed ingot iron by 23 per cent. Table VI shows that understressing plus subsequent coaxing may further increase the endurance limit. Figure 11 shows that the effect of understressing plus subsequent coaxing has a much more marked effect on endurance life than on endurance limit, and that endurance life may be increased by relatively enormous percentages.

CONCLUSIONS

1. The overstressing tests show that when a material is subjected to a stress above the endurance limit, damage begins to appear at some time during the progress of the test, which has the effect of reducing the endurance limit. Thus, a specimen which originally had an overstress of, say, 10 per cent, has its endurance limit decreased after a certain number of cycles. This reduced endurance limit means that the specimen is being subjected to an actual overstress greater than 10 per cent, which would tend to increase the percentage of damage and the rate at which damage proceeds.

2. Materials seem to vary in their ability to withstand overstress. Some materials, at certain percentages of overstress, are damaged at a comparatively slow rate, and some even exhibit recovery

from damage during part of the test. As might be expected, the higher percentages of overstress usually show greater damage and greater rate of damage.

3. Even when there is only slowly increasing damage or even recovery during part of the test, it seems probable that during the last portion of the test before failure there must be rapidly increasing damage.

4. When the overstress is not too large, say, 10 or perhaps even 20 per cent, the materials seem to be able to withstand the overstress for a large proportion of their normal life without suffering alarming percentages of damage.

5. The understressing tests show that the amount of strengthening obtained from understressing is partly determined by the number of cycles applied. However, there seems to be an upper limit of strengthening beyond which further cycles cease to increase the endurance limit.

6. After periods of understressing, various coaxing increments of stress may further increase the endurance limit.

7. The most marked effect of understressing plus coaxing is the comparatively enormous increase in endurance life that may be obtained at stresses well above the original endurance limit.

8. The tests show that both overstressing and understressing change the endurance limit. It seems clear that any stress applied to a material changes the properties of that material to a greater or lesser degree. Overstressing in fatigue requires comparatively few cycles to show this change, while understressing requires many cycles.

The author wishes to express his thanks to the American Rolling Mill Co. for furnishing the ingot iron used in the tests, and to the Carnegie-Illinois Steel Corp. for furnishing the S.A.E. 1030 steel. Thanks are due the Research Committee on Fatigue of Metals of the American Society for Testing Materials, the Wisconsin Alumni Research Foundation, and The University of Wisconsin, for furnishing the funds to carry on the testing program.

This work has been carried out as a research project in the Department of Mechanics, of which M. O. Withey is chairman, and in the College of Engineering, of which F. Ellis Johnson is dean.

DISCUSSION

MR. G. W. STICKLEY[1] (*presented in written form*).—In connection with the improvement in fatigue life of annealed ingot iron by understressing plus coaxing as reported by Mr. Kommers, it may be of interest to cite the improvement that has been observed at the Aluminum Research Laboratories in recent tests of some heat-treated wrought aluminum alloy rod. These tests differed from Mr. Kommers' tests, however, in that the specimens were subjected to moderate overstressing instead of understressing. In one of the tests, for example, the specimen first was subjected to a stress of 20,750 psi., which was 12 per cent above the endurance limit of 18,500 psi. As shown in the accompanying Table I, stress increments of 250 psi. were added, usually at intervals of 20 million cycles. The final stress was 22,000 psi. and the total number of cycles at failure about 86 million. This total number of cycles was twice the life that would have been expected if the initial stress of 20,750 psi. had been used continuously throughout the test. Even the total number of cycles, 25 million, applied at stresses of 21,750 and 22,000 psi. was equal to the normal fatigue life at 21,750 psi., despite the fact that the specimen previously had been subjected to a total of 61 million cycles at stresses from 20,750 to 21,500 psi., all above the endurance limit.

TABLE I.—RESULTS OF TEST OF OVERSTRESSED SPECIMEN.

Stress Periods	Maximum Stresses Used, psi.	Cycles, millions	Nominal Life, million cycles[a]	Ratio of Total Cycles to Nominal Life
DATA FROM INDIVIDUAL STRESS PERIODS				
1.....	20 750	20	43	0.47
2.....	21 000	20	38	0.53
3.....	21 250	20	33	0.61
4.....	21 500	1	29	0.03
5.....	21 750	20	26	0.77
6.....	22 000	5	24	0.21
SUMMARY OF RESULTS				
1 to 6...	20 750 to 22 000	86	43	2.00
2 to 6...	21 000 to 22 000	66	38	1.74
3 to 6...	21 250 to 22 000	46	33	1.39
4 to 6...	21 500 to 22 000	26	29	0.90
5 to 6...	21 750 to 22 000	25	26	0.96
6	22 000	5	24	0.21

[a] At lowest stress as indicated by regular fatigue curve determined for same lot of material.

MR. ERNEST E. THUM.[2]—I should like to make some remarks only to amateurs, because experts in fatigue testing will know what is meant when one says overstressing. I want to emphasize the fact that overstressing, which leaves residual *tension* in the external fibers of these samples, are the only ones which are dangerous. Overstressing which leaves residual compression in the surface will improve the endurance qualities. That fact, of course, is now frequently used in practice by shot blasting a finish-machined part and in that way inducing heavy compression stresses in it, and, therefore, actually increasing the margin between the stress at the surface before a load is put on it and the damaging tension stress from the load.

It is, of course, realized that the only type of stress which will cause fatigue failure is tension stress; compressive fatigue failures are impossible.

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[1] Research Metallurgical Engineer, Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[2] Editor, *Metal Progress*, American Society for Metals Cleveland, Ohio.

The intelligent designer of machine parts is usually skeptical of the utility of fatigue tests and endurance limits as determined by the rotating-beam machine. The present trend is to test a complete machine unit, as is observable in such work as Mr. Horger is doing at Canton, where complete railroad axes and complete axle-wheel assemblies are being tested for their endurance limit. The results are very much lower than given by the same material when cut up into small laboratory specimens.

The tendency also in careful design for mass production would be to design not for infinite life but for economical life. That means, therefore, that the inclined portion of the *S-N* curve is the important portion, rather than the part which is horizontal and which leads off supposedly to infinity without fracture. The tendency, I believe, in the study of complete machine parts or machine assemblies is to test in such a way as will determine cycles to failure in a limited time—get some idea of the life at full load and at certain overloads. In the study of such results a considerable scatter is expected for it is intelligent to look upon the steep part of the *S-N* curve as a mortality curve or life expectancy curve rather than as a straight line.

MR. J. B. KOMMERS[3] (*author's closure, by letter*).—I wish to thank Mr. Thum for his additional explanatory discussion. The behavior of materials under various fatigue stressing operations is first studied necessarily on small specimens in order to keep the expense of an investigation within proper limits. As Mr. Thum points out, the behavior of a complete machine unit must be determined later either by full size units in the laboratory or by operation in service.

The phenomenon of scatter, referred to by Mr. Thum, has probably not received the attention that it deserves. Since this paper was published, I have made tests in which the damage due to overstress was measured by the change in endurance life which occurred at a subsequent higher or lower overstress. Such tests show that any individual specimen develops an endurance life which may differ widely from the expected average.

The tests reported by Mr. Stickley are in line with tests which I have made and which I hope to amplify and report in a future paper. Because of the long life of aluminum alloys, these materials are particularly adapted for trying the effect of various overstressing operations. My own tests on steels show that at low cycle ratios and low overstresses there may be considerable strengthening, from the standpoint both of increased endurance limit and increased endurance life. Such operations plus coaxing should have the effect shown by Mr. Stickley's results.

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[3] Professor of Mechanics, College of Engineering, University of Wisconsin, Madison, Wis.

THE FATIGUE PROPERTIES OF SOME COLD-DRAWN NICKEL-ALLOY WIRES

By John N. Kenyon[1]

Synopsis

Fatigue data are given on eight nickel-alloy wire materials wherein the curves were carried out to 100 × 10^6 cycles. Since the tests were made on commercially drawn wire, and with fatigue failure occurring at any point within a 15-in. length, the data offer interesting comparison with those obtained with the standard rotating-beam fatigue machine. An interesting range of modulus of elasticity values are reported for the several alloys and thermal treatments.

Materials

The test specimens were commercially produced, nickel wire, cold drawn to approximately 0.0375 in. in diameter and subjected to various thermal treatments. All alloys except the iron-nickel-chromium alloy were cast in 14 by 14 by 60-in., 3100-lb. ingots; milled, chipped, and forged to 8 by 8-in. blooms; chipped, and hot rolled to 3 by 3-in. billets; chipped, and hot rolled to $\frac{1}{4}$-in. diameter wire rod; annealed, pickled, and cold drawn to wire; the final cold drawing was 75 per cent reduction of area. The iron-nickel-chromium alloy was processed similarly, except that it was cast as a 10 by 10 by 40-in., 1000-lb. ingot. See Tables I and II.

Test Procedure

The fatigue tests were performed with the rotating-wire arc fatigue machine described by the author in 1935[2] and 1940.[3]

The functioning principle of the rotating-wire arc machine is essentially that of bending a wire test specimen to a curve by means of two inclined bearing metal guides and rotating this wire from one end by a motor. The other end of the wire is left free to slide longitudinally in its guide and, in so doing, this test specimen *automatically assumes the curvature of the circular arc.* Some 15 in. of wire are thus subjected to equal stress reversals with fatigue failure occurring at the weakest point within this length. The rotating arc dips into an oil bath which dampens out vibrational effects. This dampening method offers the unique

TABLE I.—MATERIALS.

Nickel	Stress-equalized annealed at 575 F.
Monel	Stress-equalized annealed at 575 F.
Inconel	Stress-equalized annealed at 800 F.
K Monel	Stress-equalized annealed at 575 F.
K Monel	Age hardened at 1000 F. for 8 hr., and furnace cooled
Iron-Nickel-Chromium	Stress-equalized annealed at 800 F.
Nickel-Chromium-Iron	Stress-equalized annealed at 800 F.
Z Nickel	Age hardened at 900 F. for 8 hr. and furnace cooled

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[1] Research Engineer, New York, N. Y.

[2] John N. Kenyon, "The Rotating-Wire Arc Fatigue Machine for Testing Small-Diameter Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 156 (1935).

[3] John N. Kenyon, "A Corrosion-Fatigue Test to Determine the Protective Qualities of Metallic Platings," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 705 (1940).

TABLE II.—CHEMICAL COMPOSITION.

Alloy	Carbon, per cent	Manganese, per cent	Iron, per cent	Sulfur, per cent	Silicon, per cent	Copper, per cent	Nickel, per cent	Aluminum, per cent	Chromium, per cent	Molybdenum, per cent
Nickel	0.05	0.27	0.10	0.005	0.07	0.02	99.46	...	...	...
Monel	0.12	0.97	1.06	0.019	0.04	31.42	66.35	...	...	...
Inconel	0.12	0.19	6.38	0.011	0.29	0.11	79.60	...	13.28	...
K Monel	0.15	0.47	0.61	0.005	0.24	29.16	65.75	3.04	...	...
Iron-Nickel-Chromium	0.12	0.70	54.81	0.005	0.34	0.11	35.98	...	7.36	0.55
Nickel-Chromium-Iron	0.05	0.22	6.58	0.009	0.45	0.05	77.43	...	13.19	...
Z Nickel	0.23	0.20	0.19	0.005	0.20	0.02	98.45	...	...	...

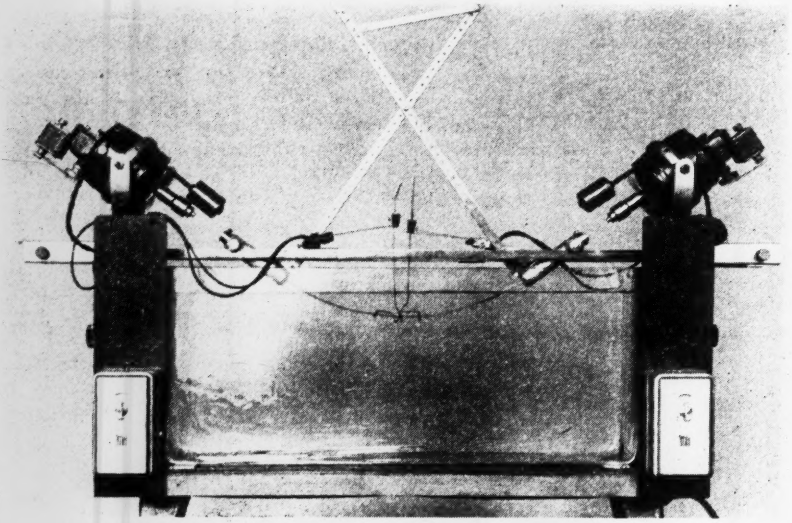

FIG. 1.—The Rotating-Wire Arc Fatigue Machine.

Note.—Two wire specimens are being tested simultaneously, but in this photograph appear as one wire. Time exposure 15 sec. with the motors operating at 5000 rpm.

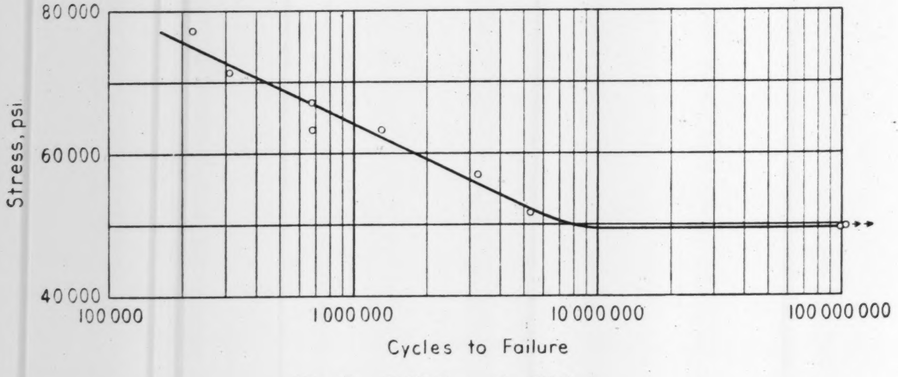

FIG. 2.—*S–N* Curve for Nickel.

advantage for making corrosion-fatigue tests, wherein the oil bath is replaced with various corrosive solutions (Fig. 1).

As the fatigue tests were carried out in a highly neutral oil bath, it is believed that further valuable information may be obtained by employing corrosive media. The following are being contemplated:

1. Sulfuric acid (5 per cent solution).
2. Sea water (U. S. Navy Standard Solution).
3. Carbonated water (Carbon dioxide, saturated).
4. "Sour" oil (hydrogen sulfide, saturated).

TABLE III.—MECHANICAL PROPERTIES.

Material	Elongation in 2 in., per cent[a]	Modulus of Elasticity, psi.	Tensile Strength, psi.	Fatigue Limit, psi.	Ratio of Fatigue Limit to Tensile Strength
Nickel	2.75	31 200 000	144 200	49 500	0.343
Monel	3.00	28 100 000	140 000	46 500	0.332
Inconel	5.30	34 000 000	207 000	50 500	0.244
K Monel (stress-equalized)	2.00	26 300 000	162 000	48 500	0.299
K Monel (age-hardened)	2.50	27 400 000	200 000	44 500	0.223
Iron-Nickel-Chromium	3.50	30 100 000	151 000	45 500	0.302
Nickel-Chromium-Iron	3.00	33 200 000	201 000	49 000	0.244
Z Nickel	8.00	34 000 000	231 500	42 500	0.184
Steel	...	30 200 000	(Modulus control material)		

[a] Average of three or more tests determined on other wires than those used for the tensile strength and modulus of elasticity determinations.

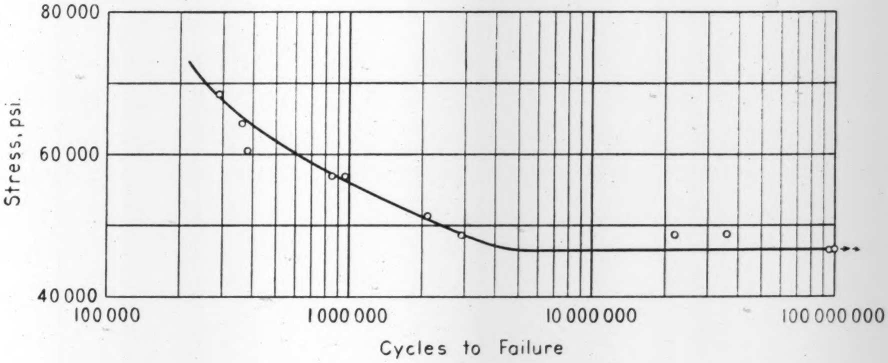

FIG. 3.—*S-N* Curve for Monel.

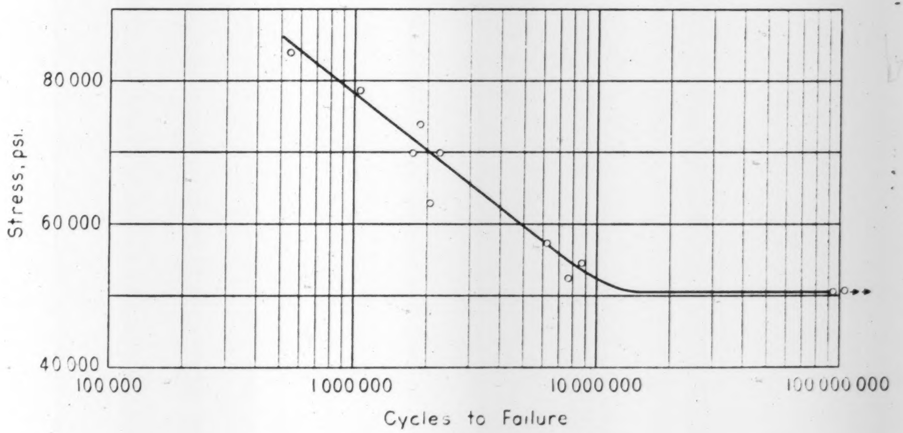

FIG. 4.—*S-N* Curve for Inconel.

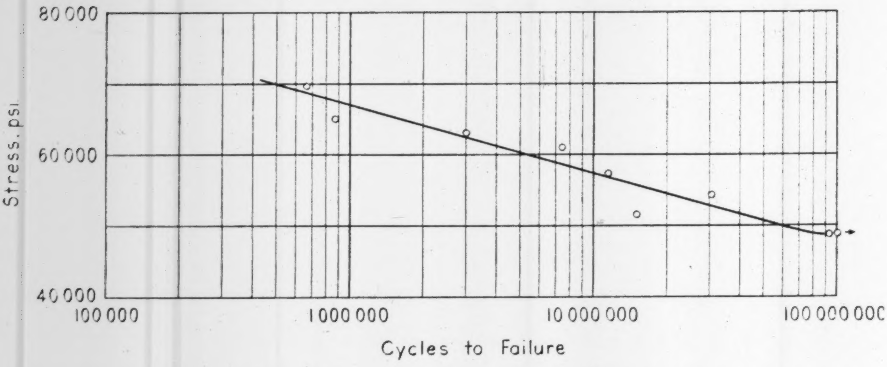

FIG. 5.—*S-N* Curve for K Monel (Stress-Equalized).

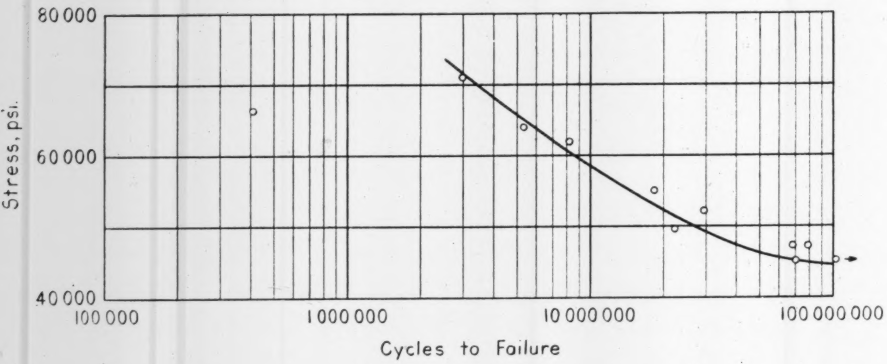

FIG. 6.—*S-N* Curve for K Monel (Age-Hardened).

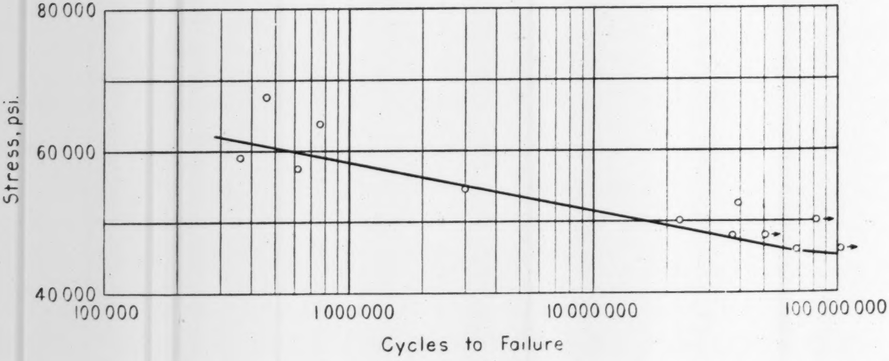

FIG. 7.—*S-N* Curve for Iron-Nickel-Chromium.

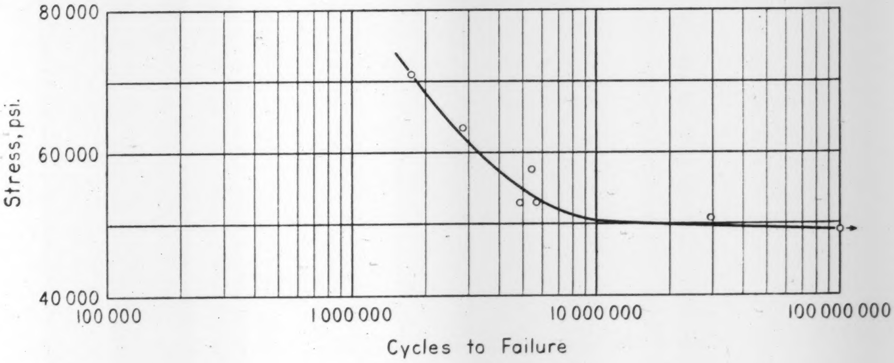

FIG. 8.—*S-N* Curve for Nickel-Chromium-Iron.

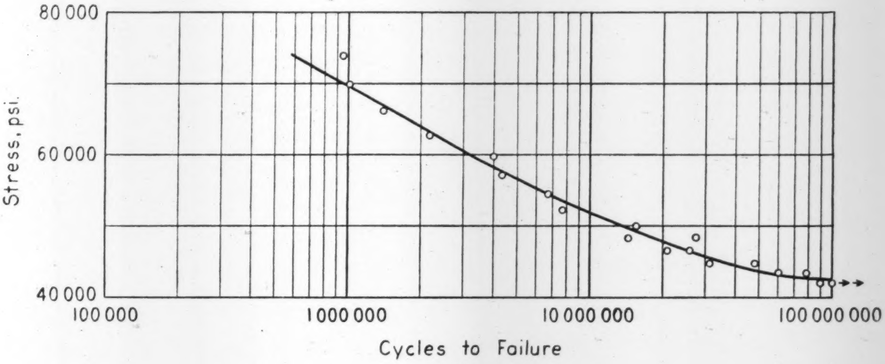

FIG. 9.—*S-N* Curve for Z Nickel.

TABLE IV.—MODULUS OF ELASTICITY VALUES.

Nickel	Monel	Inconel	K Monel		Iron-Nickel-Chromium	Nickel-Chromium-Iron	Z Nickel	Steel
			Stress-equalized	Age-hardened				
31 700 000	28 000 000	33 100 000	26 500 000	27 400 000	30 100 000	33 500 000	33 300 000	30 100 000
31 400 000	28 200 000	34 500 000	26 000 000	26 000 000	30 000 000	33 800 000	33 700 000	30 300 000
30 800 000	28 000 000	34 200 000	26 000 000	27 800 000	29 700 000	34 000 000	33 500 000	30 400 000
30 900 000	27 900 000	34 600 000	26 300 000	27 900 000	30 300 000	32 500 000	34 000 000	29 900 000
31 200 000	28 500 000	33 700 000	26 600 000	27 900 000	30 300 000	32 300 000	34 900 000	30 400 000
31 200 000	28 100 000	34 000 000	26 300 000	27 400 000	30 100 000	33 200 000	34 000 000	30 200 000

SUMMARY OF RESULTS

The fatigue data are summarized in Table III and in Figs. 2 to 9.

No fatigue values were excluded from the data except where an irregularity could be attributed to mechanical accident. It is believed that all data should be reported since the regularity with which the points fall on the curves is a criterion of the uniformity of the materials. The high degree of reproducibility is well illustrated in Fig. 9 where the tests were run in duplicate on two machines.

The wire materials, commercially produced, were tested in the processed condition with the usual run of surface irregularities. Fatigue breaks were free to occur at the weakest point within a 15-in. length. These data should offer an interesting study for comparison with similar data obtained on the standard rotating-beam machine.

The data in Table IV are introduced to show the wide range of modulus of elasticity (E) values obtained on these cold-drawn nickel alloys. The modulus determinations on 0.66 carbon, cold-drawn steel wire were interspersed with those on the nickel alloys as a means of control on the accuracy of this test procedure. Modulus values higher than those commonly attributed to steel (that is, 30,000,000 psi.) have always aroused scientific interest.

Acknowledgments:

Acknowledgment is made to The International Nickel Co., Inc., and to The Alloy Metal Co., for furnishing the spring materials for the investigation; also to the Department of Civil Engineering, Columbia University, for the use of their laboratory facilities.

DISCUSSION

Mr. B. B. Betty[1] (*presented in written form*).—Mr. Kenyon has presented to the Society the results of some very carefully conducted tests of nickel alloy wires with the commercial surfaces left intact. Here again one is confronted with an old question familiar to the test engineer, namely, "to what degree is the endurance limit of such a product due to the inherent fatigue strength of the material in the core of the wire and to what extent is it influenced by the condition of the surface." It is well known that the endurance limit of a metal increases with an increase in the amount of cold work, that is, the percentage of cold reduction. Furthermore, the increase in the endurance limit is approximately proportional to the increase in tensile strength.

Advantage is being taken of this fact to estimate the endurance limits of the polished condition of these wires from rotating-beam fatigue tests of polished specimens of corresponding rod stock. From these data, then, an estimate is made of the stress concentration effect of the commercial cold-drawn surfaces. The writer has found in his records of fatigue tests, data on each of the alloys tested by Mr. Kenyon. Except in one case, however, they do not represent the same melts. They were obtained from polished specimens made from rod stock which was subjected to the same thermal treatment as were the corresponding alloys in Mr. Kenyon's work. Tests were conducted on the familiar rotating-beam machine developed by R. R. Moore, and endurance limits were determined at one hundred million cycles of stress.

The accompanying Table I lists the thermal and mechanical condition of the rod stock as well as its tensile and fatigue properties. The endurance ratios shown in the last column of this table are the ratios of the endurance limits to their corresponding tensile strengths. The fifth column of this table shows that the rod stock was given much less cold reduction than the 75 per cent used in the small wire. Hence the wire products tested by the author would have much higher tensile strengths than the rod stock. We can put these on a comparable basis, however, if we assume that polished specimens of wire should have the same endurance ratios as polshed specimens of rod stock.

In Table II of this discussion the tensile strength of the wire tested by the author is shown multiplied by the endurance ratios of the polished rod specimens to obtain an estimated endurance limit of the polished wire. These estimated endurance limits listed in column 4 are compared with the actual endurance limits found for the commercial drawn wire. The last column of this table shows the estimated percentage reduction in fatigue strength by the cold-drawn surfaces.

It should be noted that the effect of the cold-drawn surface is to lower the endurance limit roughly one third to three eighths below that of corresponding polished surfaces.

In line with the above observations

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[1] Research Engineer, The International Nickel Co., Inc., Huntington, W. Va.

TABLE I.—FATIGUE DATA ON POLISHED ROTATING BEAM SPECIMENS.

Alloy	Melt	Stock Size, in.	Condition	Cold Reduction, per cent	Yield Strength, 0.2 per cent Offset, psi.	Tensile Strength, psi.	Endurance Limit, psi,	Endurance Ratio
Nickel	N-3431-A	½	CD + S.E.A.	21	81 000	97 000	36 500	0.38
Monel	M-4808-B	½	CD + S.E.A.	21	111 000	117 000	49 750	0.42
Inconel	NX-4446	½	CD + S.E.A.	42	159 000	164 500	60 500	0.37
K Monel	M-1016-K	⅞	CD + S.E.A.	13	94 500	110 500	46 500	0.42
K Monel	M-947-K (Aged)	½	CD + Aged	21	138 500	165 500	59 000	0.36
Fe—Ni—Cr	Y-1424	½	As CD	35	142 000	154 000	56 000	0.36
Ni—Cr—Fe	NX-6063	1-¼	As CD	25	138 000	145 000	49 500	0.34
Z Nickel	N-5354-Z	⅞	CD + Aged	13	161 000	195 000	59 000	0.30

THERMAL TREATMENTS

Nickel	Stress equalized annealed 525 F., 3 hr.
Monel	Stress equalized annealed 525 F., 3 hr.
Inconel	Stress equalized annealed 800 F., 3 hr.
K Monel—M-1016-K	Stress equalized annealed 525 F., 3 hr.
K Monel—M-947-K	Aged 1070 F. for 16 hr. and cooled in furnace 25 F. per hr.
Fe—Ni—Cr	None
Ni—Cr—Fe	None
Z Nickel	Aged 930 F. for 16 hr.

are the results of some recent experiments the writer has conducted, in which the effect of the surface of hot-rolled Inconel rods was determined. In this case a rotating strut type of machine was used to test the material as hot-rolled rods. This is another type of machine in which it is not necessary to have a reduced section in the specimen in order to avoid failure at the grips. Three melts of ⅜ in. diameter hot-rolled rods were thus investigated. From each of these materials specimens for polished rotating-beam tests were cut also. The accompanying Table III lists the results of these experiments.

On comparing the results shown in Tables II and III it may be observed that surface imperfections in cold-drawn wire reduce the fatigue strength about the same amount as do those in hot-rolled rods. These may be dealt with as stress-raisers having stress concentration factors of 1.3 to 1.4. It is gratifying to find that this factor is not greater than this.

TABLE II.—ESTIMATED EFFECT OF COMMERCIAL SURFACE ON FATIGUE OF COLD-DRAWN WIRE.

Alloy	Tensile Strength of Wire Used by Kenyon, psi.	Endurance Ratio of Polished Specimens	Estimated Endurance Limit of Polished Wire, psi.	Actual Endurance Limit of Cold-Drawn Wire Found by Kenyon, psi.	Estimated Reduction in Fatigue Strength by Cold-Drawn Surface, per cent
Nickel	144 200	0.38	54 800	49 500	9.7
Monel	140 000	0.42	58 800	46 500	21
Inconel	207 000	0.37	76 600	50 500	34
K Monel	162 000	0.42	68 000	48 500	28.7
K Monel	200 000	0.36	72 000	44 500	38.2
Fe—Ni—Cr	151 000	0.36	54 300	45 500	16.4
Ni—Cr—Fe	201 000	0.34	68 500	49 000	28.5
Z Nickel	231 500	0.30	69 500	42 500	39

MR. JOHN N. KENYON[2] (*author's closure, by letter*).—Mr. Betty has given us a consistent picture of the lowering effect of surface irregularity on the endurance limit of these nickel alloys. This type of information should be of value to the designing engineer.

TABLE III.

Melt	Endurance Limit with Polished Surface, psi.	Endurance Limit with Hot-Rolled Surface, psi.	Reduction in Endurance Limit by Hot-Rolled Surface, per cent
NX6159	53 000	43 500	18
NX6166	55 500	42 000	24
NX6462	51 000	38 000	25

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[2] Research Engineer, New York, N. Y.

Perhaps of equal importance to the engineer is some information on the homegeneity; that is, just what is the probability of encountering abnormally low fatigue values in a given length of material. To complete this study, a total of 115 ft. of wire was subjected to the fatigue effect and no data rejected except where the low value could be attributed to mechanical accident. In this total of 115 ft. of wire only one abnormally low value was encountered as shown in the plotted data of Fig. 6.

FATIGUE TESTS ON SOME COPPER ALLOYS IN WIRE FORM

By H. L. Burghoff[1] and A. I. Blank[1]

Synopsis

The results of rotating-arc fatigue tests upon several copper alloys in the form of hard-drawn wire, 0.072 in. in diameter, are presented in this paper. The alloys include four brasses or copper-zinc alloys, two copper-tin alloys, two tin-bearing brasses, two silicon bronzes, four copper-nickel alloys, and two age-hardened and drawn alloys containing copper, nickel, and phosphorus.

For the brasses, endurance limit is maximum at or in the vicinity of 20 per cent zinc. Tin, nickel, and silicon enhance the endurance properties of the alloys in varying degrees, and in some instances small additions of alloying elements appear to be as effective as larger amounts. An age-hardened and severely drawn copper-nickel-phosphorus alloy has particularly attractive properties. The feasibility of several alloys, high with respect to endurance strength, but low with respect to content of critical alloying elements as compared with the commonly used phosphor bronzes, is indicated.

An approximate relationship, which indicates that the endurance limit may be expected to be high with respect to tensile strength if the yield strength, as variously determined, is high with respect to tensile strength, appears to exist for the materials of the investigation.

This paper presents the results of fatigue tests on wire of several copper alloys. The desirability of such information seems evident in view of the numerous applications of springs and other articles formed from wire and subjected to cyclic stresses.

Very little work has previously been published upon fatigue testing of copper alloys in the form of small wire, although Kenyon (**1**)[2] described a very suitable testing machine in 1935 and later presented some results obtained with it upon copper wires (**2**). Such testing of copper alloys has largely been conducted upon material in strip or rod form, the most recent contribution being that of Anderson and Smith (**3**), who performed rotating-beam tests upon rod materials. Since Kenyon, and Anderson and Smith have so recently summarized the existing literature upon endurance of copper alloys, it seems unnecessary to do so here.

Test Procedure

The fatigue tests were conducted with two rotating-wire arc machines of the type described by Kenyon (**1**). In these machines the wire specimen is gripped in a chuck mounted on the shaft of the driving motor, is passed through a bushing rigidly aligned with the motor shaft, and then is passed through a second bushing. The test section lies between these bushings. They are in alignment when disposed horizontally and may be inclined in a vertical plane through their axes. When they are inclined toward each other by an equal amount, as is done in making the tests, the specimen is deflected in what is essentially the arc of a circle, so that the bending stresses are substantially uniform along the test section and fractures may occur at random along the arc. As there must be some clearance between bushing and specimen, there is a slight departure from a true arc at this point, so that stress at the bushings is slightly lowered and fractures rarely occur at these points. Speed of rotation of the machines is 3450 rpm.

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[1] Research Metallurgist, and Assistant Research Metallurgist, respectively, Chase Brass and Copper Co., Waterbury, Conn.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 783.

Maximum fiber stress is calculated from the formula:

$$S = \frac{EI}{RZ} = \frac{Ed}{2R} = \frac{Ed}{C} \sin \Theta$$

where:

S = stress,
E = modulus of elasticity,
I = moment of inertia,
Z = section modulus,
d = diameter of wire specimen,
R = radius of curvature,
Θ = angle of inclined bushing with the horizontal, and
C = horizontal chord = $2\,R \sin \Theta$.

It will be noted that, for a given material, stress may be varied by adjustment of either or both the chord length and the angle of inclination with respect to the horizontal. In the tests reported here it was found convenient to maintain the angle constant at 30 deg. and to obtain the desired range of stress by adjustment of the chord length.

The calculation of stress also requires a knowledge of the modulus of elasticity of the test material. As there is deviation from proportionality in the stress-strain relationship for most copper alloys, particularly at the highest stresses employed in these fatigue tests, a single value of modulus cannot be used; the stress-strain relationship through the desired range must be obtained experimentally. This was accomplished in the present investigation by means of a Templin-type automatic stress-strain recorder used in conjunction with a 1940 model Baldwin-Southwark testing machine of high sensitivity. The actual determination of the proper value of modulus is readily made by trial for the particular setting of the fatigue testing machine.

The possibility of an effect of the cyclic stressing of the wire during the fatigue tests upon the stress-strain relationships must naturally be considered. To obtain information on this point, stress-strain curves were obtained for 70–30 copper-zinc wire, drawn 60 and 84 per cent, both before and after being subjected to an alternating maximum fiber stress of approximately 34,000 psi. for 100,000 and 250,000 cycles. No distinguishable difference could be found in the curves for these materials before or after subjection to the alternating stresses. It is believed that the other materials tested in the drawn condition behave in a similar manner over the range of stresses encountered in the fatigue testing and that, therefore, the simple stress-strain curves are properly applicable.

MATERIALS

The materials whose compositions, tempers, and properties are listed in Table I were prepared and tested in the form of wire, 0.072 in. in diameter. Most of them were finished in commercial hard and spring tempers, that is, as drawn 60 and 84 per cent, respectively, after the last anneal. As all were drawn in coil form, it was necessary to straighten them mechanically before testing. Practically perfect straightness of the fatigue specimens is required in order to avoid the formation of standing waves which affect the stress. It is to be remarked that the straightening operation, which was accomplished in a commercial machine with rotating-die assembly, results in a reduction of tensile strength which amounts to approximately 10 per cent of the strength of the material as drawn in coil form. There is a small corresponding increase in elongation value. All properties reported in this paper are for straightened material of commercial surface finish, as tested in air at ordinary temperature.

TABLE I.—COMPOSITIONS, TEMPERS, AND PROPERTIES OF ALLOYS INVESTIGATED.

Alloy Composition, per cent	Temper, per cent reduction	Stress at 0.01 per cent Offset, psi.	Stress at 0.2 per cent Offset, psi.	Stress at 0.5 per cent Extension, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Stress for life of 1,000,000 Cycles, psi.	Endurance Limit for 100,000,000 Cycles, psi.	Ratio, Endurance Limit to Tensile Strength
91.20 Cu, 8.80 Zn	84	30 000	63 000	61 500	72 500	4.0	31 500	23 000	0.32
	60	29 000	53 500	54 000	61 000	5.5	29 000	21 000	0.35
80.06 Cu, 19.94 Zn	84	33 500	80 500	66 500	108 000	3.5	37 000	26 500	0.25
	60	30 500	67 500	61 000	89 000	7.5	35 000	23 000	0.26
69.32 Cu, 30.65 Zn	84	35 000	85 500	65 500	120 000	3.5	36 500	22 000	0.18
	60	28 000	66 500	62 500	98 500	5.0	35 500	19 500	0.20
62.62 Cu, 37.38 Zn	84	28 500	82 000	62 500	118 000	3.0	36 500	22 000	0.19
	60	32 000	66 000	60 000	93 000	5.5	35 000	19 000	0.20
98.79 Cu, 1.20 Sn	84	32 500	62 500	63 500	72 000	9.0	39 500	32 000	0.45
	60	32 500	55 500	56 500	66 000	11.0	37 500	28 500	0.43
95.41 Cu, 4.37 Sn, 0.30 P	84	48 000	101 500	80 500	121 000	5.0	45 000	30 000	0.25
79.90 Cu, 0.79 Sn, 19.30 Zn	84	35 000	93 000	70 000	125 000	4.0	43 000	34 500	0.28
	60	30 500	79 000	68 000	104 000	5.0	40 000	28 500	0.27
71.39 Cu, 0.92 Sn, 27.67 Zn	84	32 500	92 000	69 500	133 000	1.5	41 000	26 500	0.20
	60	32 500	80 500	69 000	106 500	3.0	39 000	22 500	0.21
97.12 Cu, 1.39 Si, 1.45 Zn	84	33 000	75 500	68 000	93 500	5.0	36 000	28 000	0.30
	60	27 000	57 500	57 000	73 000	7.0	33 000	24 500	0.34
96.22 Cu, 2.82 Si, 0.95 Zn	80	28 500	89 500	67 000	133 500	4.0	42 500	29 500	0.22
	60	28 500	77 500	62 000	111 000	5.5	40 000	28 500	0.26
89.48 Cu, 10.08 Ni, 0.49 Mn	88	37 000	67 000	66 500	69 500	5.5	35 000	29 000	0.42
79.53 Cu, 20.02 Ni, 0.46 Mn	88	34 500	78 000	76 500	84 300	4.5	41 000	34 000	0.40
75.82 Cu, 23.38 Ni, 0.48 Mn	88	36 500	84 000	82 000	93 000	5.0	44 500	39 000	0.42
70.24 Cu, 29.11 Ni, 0.50 Mn	88	35 500	89 500	86 000	96 200	4.0	44 500	35 000	0.36
98.59 Cu, 1.13 Ni, 0.21 P	98	49 000	103 500	87 000	116 000	3.5	44 000	40 000	0.35
	94	52 000	96 500	84 500	109 000	4.5	44 000	39 000	0.36
	75	40 000	82 500	76 500	91 000	5.5	42 000	33 500	0.37
97.94 Cu, 1.10 Ni, 0.75 Sn, 0.22 P	94	53 000	109 000	86 000	124 000	2.5	44 500	38 500	0.31

DISCUSSION

The stress-strain curves used in the calculation of maximum fiber stresses developed in the wire specimens during the fatigue tests are shown in Figs. 1 to 5. They require little comment other than to point out once more the nonlinear relationship, particularly at many of the higher stresses used in the fatigue tests.

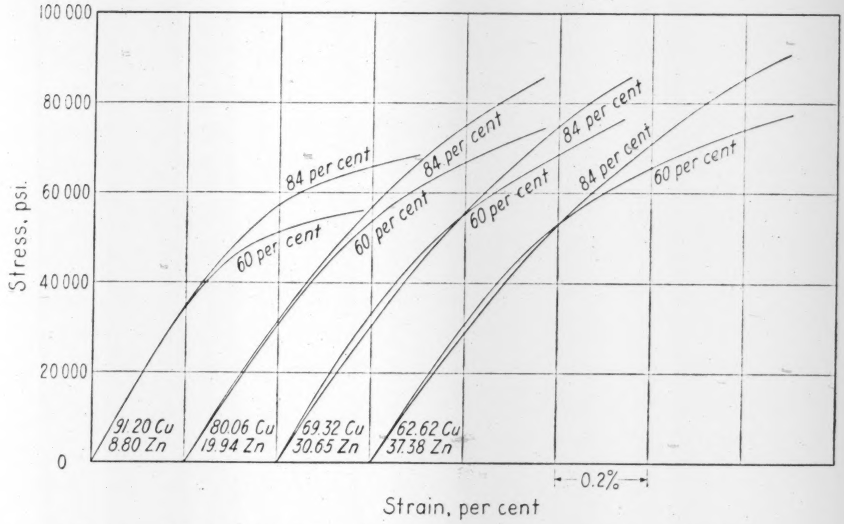

FIG. 1.—Stress-Strain Curves for Copper-Zinc Alloys Drawn as Indicated.

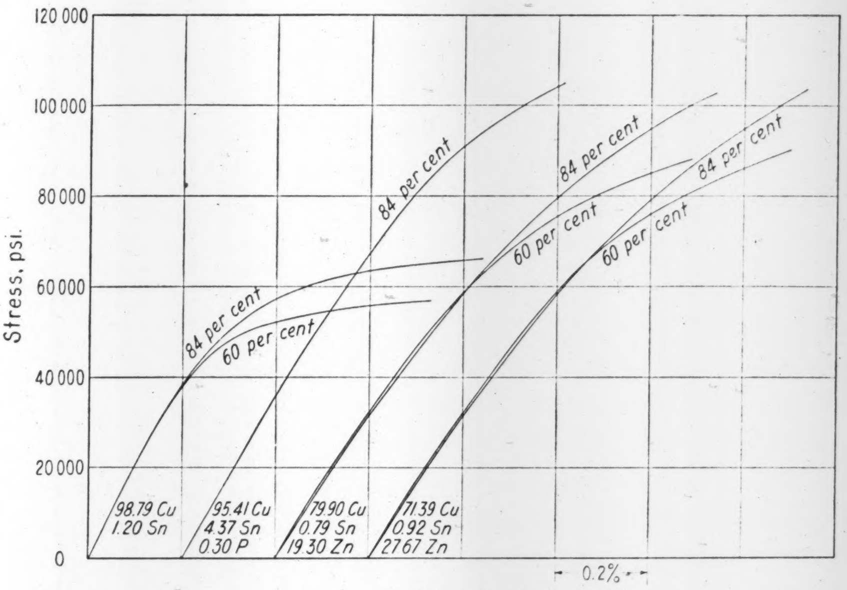

FIG. 2.—Stress-Strain Curves for Tin Bronzes and Tin-Bearing Brasses, Drawn as Indicated.

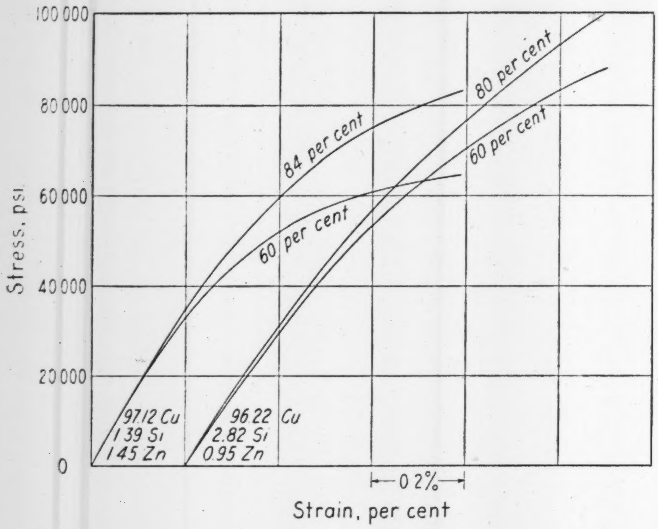

FIG. 3.—Stress-Strain Curves for Silicon Bronzes, Drawn as Indicated.

The fatigue test data are given in the *S-N* curves in Figs. 6 to 10. In every case the curves are carried out to 100,000,000 cycles or reversals of stress. The endurance limits listed in Table I are the values of stress taken from the curves at 100,000,000 cycles.

The effect of the temper of the several alloys is consistent in that endurance limit increases, although not proportionately, as tensile strength increases. In no case is there a decrease of endurance limit of an alloy for a more severely drawn condition.

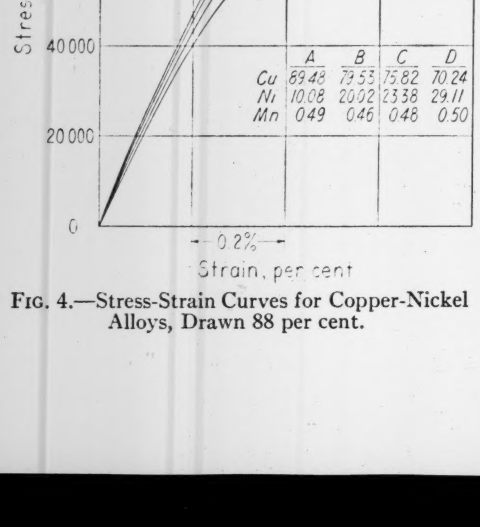

FIG. 4.—Stress-Strain Curves for Copper-Nickel Alloys, Drawn 88 per cent.

The endurance curves for the copper-zinc alloys are given in Fig. 6. The effect of zinc upon the endurance limit is not a simple function of the increasing zinc content because maximum endurance limit for the two tempers involved is attained at or in the vicinity of 20 per cent zinc. Endurance limit is not a simple function of tensile strength. The tensile strength of the 80–20 alloy, which has the highest endurance limit of this group, is slightly less than that of the 70–30 and 63–37 alloys. Tensile strength of the 91–9 alloy, which has the second highest endurance limit of the group, is by far the lowest. The ratio of endurance limit to tensile strength as shown in Table I decreases substantially as zinc content increases, however.

It is interesting to note that the slope of the upper portion of the *S-N* curve increases as zinc content increases.

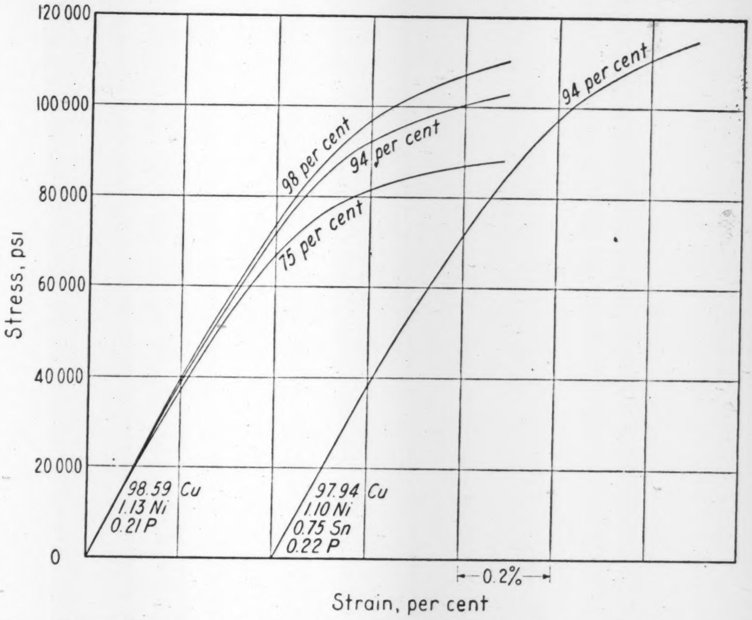

FIG. 5.—Stress-Strain Curves for Copper-Nickel-Phosphorus and Copper-Nickel-Phosphorus-Tin Alloys, Age-Hardened and Drawn as Indicated.

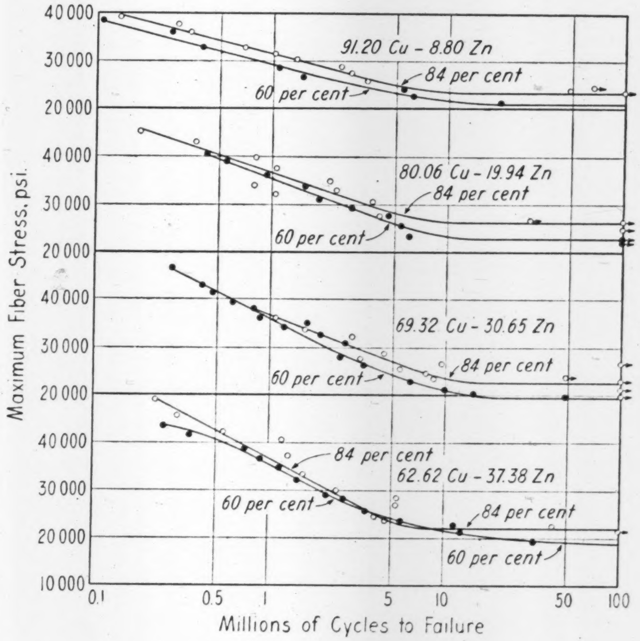

FIG. 6.—*S-N* Curves for Copper-Zinc Alloys.
Arrows indicate unbroken specimens.

This indicates that the relative fatigue resistance of the alloys of higher zinc content improves at stresses above the endurance limit and may be illustrated by a comparison of the stresses yielding a life of only 1,000,000 cycles as shown in Table I. For this life the fatigue strengths of the 80–20, 70–30, and 63–37 alloys are essentially the same, while that of the 91–9 alloy is considerably lower.

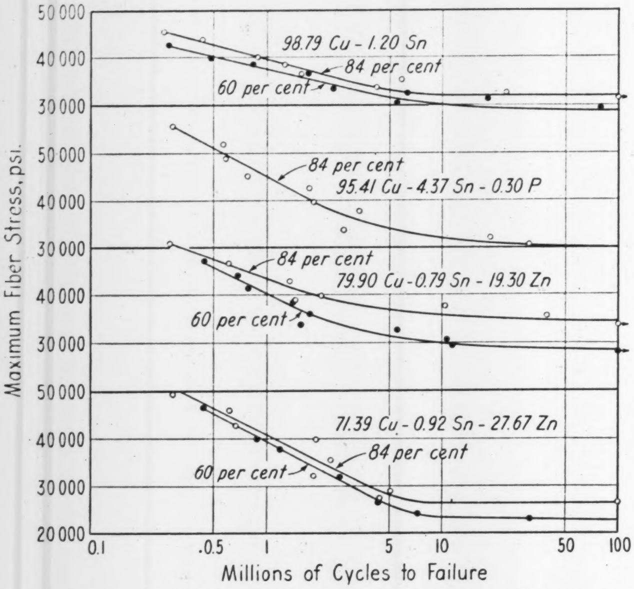

FIG. 7.—*S-N* Curves for Tin Bronzes and Tin-Bearing Brasses.
Arrows indicate unbroken specimens.

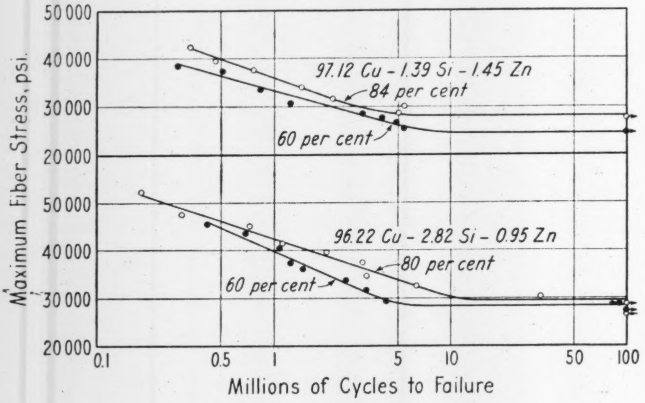

FIG. 8.—*S-N* Curves for Silicon Bronzes.
Arrows indicate unbroken specimens.

The two tin bronzes do not offer a strictly true evaluation of the effect of tin content because of the presence of 0.30 per cent phosphorus in the alloy of higher tin content. However, it does appear that only small additions of tin to copper are required to attain a relatively high endurance limit (Fig. 7). This confirms the results obtained by Anderson and Smith (3) in tests upon copper-tin alloys in rod form. The endurance limit of the 1.2 per cent tin alloy as drawn 84 per cent is actually higher than that of the 4.37 per cent tin, 0.30 per cent phosphorus alloy, but the stress which produces failure at 1,000,000 cycles is distinctly higher for the alloy of higher tin content. This fatigue strength at 1,000,000 cycles, 45,000 psi., is exceeded by no other material reported in this paper.

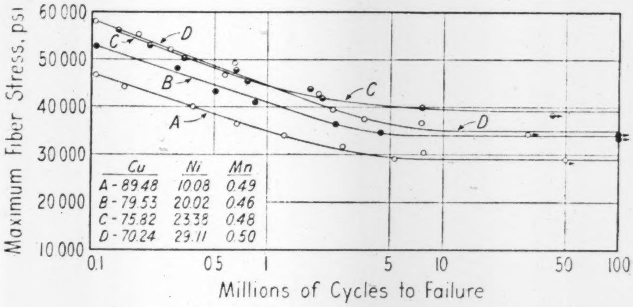

FIG. 9.—*S-N* Curves for Copper-Nickel Alloys.
Arrows indicate unbroken specimens.

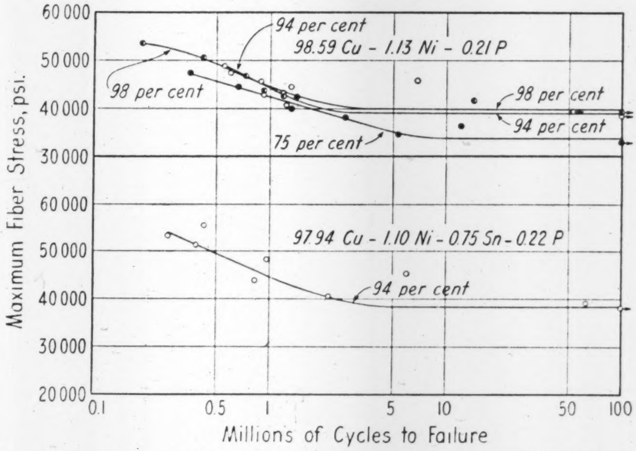

FIG. 10.—*S-N* Curves for Copper-Nickel-Phosphorus and Copper-Nickel-Phosphorus-Tin Alloys.
Arrows indicate unbroken specimens.

Figure 7 also shows the effect of 1 per cent tin in 80–20 and 70–30 brasses. The resistance to fatigue is increased by the tin, but to a much greater extent in the 80–20 alloy than in the 70–30 alloy. The endurance limit of the 80 per cent copper alloy is definitely higher than that of the bronze containing 4.37 per cent tin, and its fatigue strength for 1,000,000 cycles is only slightly lower—43,000 psi.

Silicon, like tin, markedly increases the endurance limit of copper (Fig. 8). The 3 per cent silicon bronze as drawn 80 per cent appears to be but slightly, if at all, inferior at 100,000,000 cycles to the 4.37 per cent tin bronze wire. The endurance limit of the 1.5 per cent silicon bronze as drawn 84 per cent is almost as high as that for the 3 per cent silicon bronze drawn 80 per cent. The difference, in favor of the 3 per cent silicon alloy, is more marked as drawn 60 per cent.

The *S-N* curves for four copper-nickel alloys as drawn 88 per cent are shown in Fig. 9. These alloys had originally been prepared as 0.081 in. diameter wires for another investigation in our laboratory (**4**), and were redrawn hard to 0.072 in. diameter for the present tests. Although nickel is not a very intense hardener of copper, it is seen to be very effective in increasing fatigue resistance. The test results indicate that the alloy containing 23 per cent nickel may be one of critical composition, for its endurance limit is considerably greater than for either the 80–20 or 70–30 alloys.

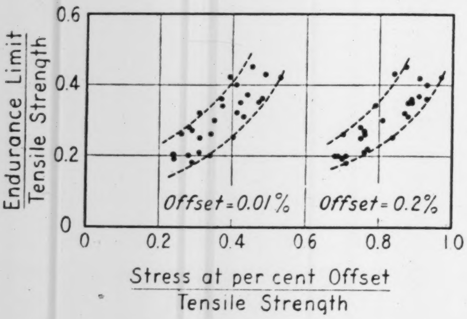

FIG. 11.—Relationships Between the Ratio of Endurance Limit to Tensile Strength and the Ratios of the Stresses at 0.01 and 0.2 per cent Offsets to Tensile Strength for all the Materials Included in the Investigation.

Among the most interesting of the alloys studied is that containing copper, nickel, and phosphorus (Fig. 10). This is an age-hardenable alloy of relatively high electrical and thermal conductivity, for which advantage has been taken of the fact that the strengthening imparted by age-hardening may be greatly augmented by subsequent cold working. Its general characteristics have been described elsewhere (**4, 5**). It was finished for testing in this program by quenching from 1450 F., aging 4 hr. at 850 F., and then cold-drawing 75, 94, and 98 per cent. The endurance limits attained for these treatments are outstanding for copper alloys of similar tensile strength.

As age-hardened and drawn 94 per cent, the copper-nickel-phosphorus-tin alloy has essentially the same endurance limit as the above alloy, from which it differs in composition by the addition of 0.75 per cent tin. In this case, the addition of tin has not affected fatigue resistance, although it has improved the ordinary tensile properties.

Correlation of fatigue properties of an alloy with other more readily obtained properties is always a tempting subject, but it is quite well known, and the data presented here serve as additional confirmation, that the tensile properties of copper alloys in general do not provide an index of endurance limit. Neither the tensile strength nor the stress at any particular offset or extension appear to be associated with the endurance limit of a material, nor does there appear to be any clear relationship between individual tensile properties and endurance properties.

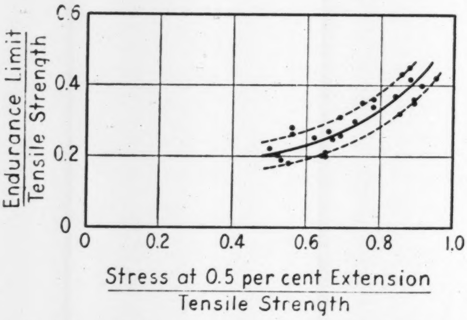

FIG. 12.—Relationship Between the Ratio of Endurance Limit to Tensile Strength and the Ratio of the Stress at 0.5 per cent Extension to Tensile Strength for all the Materials Included in the Investigation.

Figures 11 and 12 demonstrate, however, that the ratio of endurance limit to tensile strength is a rather definite function of any one of several ratios involving tensile stresses at particular offsets or extension and the tensile strength. The choice of the ratios for the abscissas in these figures is purely arbitrary, and it is likely that the ratios derived from stresses at different offsets or extensions would show a similar relationship with the ratio of endurance limit to tensile strength. The relationship is such as to indicate that endurance limit may be expected to be high with respect to tensile strength if the yield strength, determined as the stress at some offset or extension, is high with respect to tensile strength.

Of the three combinations illustrated, that in Fig. 12 shows the least scatter. The maximum deviations of the experimentally derived points from the mean curve in this figure represent plus or minus deviations in endurance limit of about 15 per cent at the upper end of the curve and about 20 per cent at the lower end.

This relationship between the ratio of endurance limit to tensile strength and the ratio of stress at 0.5 per cent extension to tensile strength might conceivably be used to estimate with 15 or 20 per cent accuracy the endurance limits of copper-base alloys of the general types included in this investigation. The tensile properties involved are more readily obtained than the *S-N* curve. Therefore, preliminary calculation of the approximate endurance limit might thus serve as a guide in selecting materials for extensive fatigue tests, but, since the method is derived from tests on severely drawn and mechanically straightened wire of small diameter, its application should perhaps be limited to material of that type.

SUMMARY AND CONCLUSIONS

Of the numerous wire materials tested in this investigation, several have been found to have endurance limits ranging from approximately 30,000 to 40,000 psi. at 100,000,000 cycles. Tin, nickel, and silicon enhance the endurance properties of these copper alloys in varying degrees, and in some instances small additions of these elements appear to be as effective as larger amounts. An age-hardened and severely drawn copper-nickel-phosphorus alloy has particularly attractive properties. All these results indicate the feasibility of several alloys, high with respect to endurance strength, but low with respect to content of critical alloying elements as compared with the commonly used phosphor bronzes.

An approximate relationship between the ratio of endurance limit to tensile strength and the ratio of yield strength, as variously determined, to tensile strength appears to exist for the materials of the investigation.

Acknowledgments:

The authors wish to make due acknowledgment to D. K. Crampton for his interest and encouragement in the investigation, to E. C. Bohlen, who made some of the stress-strain determinations, and to the Chase Brass and Copper Co. for permission to publish the results.

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DISCUSSION

Mr. John N. Kenyon.[1]—It is interesting that two papers should be presented at this meeting giving endurance data on two important non-ferrous alloyed wire materials (copper and nickel). The fact that the arc method was used for obtaining these data offers an opportunity for comparing relative values.

The arc method is unique in that it subjects a considerable length of material to the endurance test, therefore it would add to this paper if the authors can estimate the length of wire used to establish the various *S-N* curves. This information together with the regularity with which the points fall on the curves will give good indication of homogeneity. It is important for the engineer to know the order of structural uniformity in processed materials.

Messrs. H. L. Burghoff[2] and A. I. Blank[2] (*authors' closure, by letter*).—Some twenty or thirty feet of wire are necessary to establish each *S-N* curve. Nonuniformity due to structural or surface variations becomes very evident in the degree of scatter of fatigue results. Surface markings resulting from poor straightening or finishing operations can be the cause of serious deviations of individual results from the mean *S-N* curve. Limited scatter of results might therefore well serve as an indication of uniformity.

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[2] Research Metallurgist, and Assistant Research Metallurgist, respectively, Chase Brass and Copper Co., Waterbury, Conn.

PREFERRED ORIENTATIONS IN ROLLED METALS: CONSTRUCTION OF POLE FIGURES

By Beulah Field Decker[1]

It is known that many of the properties of worked metals are influenced by the orientations of the crystalline grains within the metal. Therefore, in an investigation of worked metals, it is desirable to know whether any preferment in orientation exists and also the extent and nature of that preferment. In drawn wire, because of the nature of the working—identical treatment in all directions perpendicular to the wire—preferment is shown only along the axis of the wire, with all possible orientations present in directions perpendicular to the wire; that is, a uni-directional preferment exists. However, for rolled sheet metal, with which this paper is concerned, the treatment is not identical for all directions perpendicular to the rolling direction, and consequently there is preferment shown with respect to two directions—the rolling direction and the cross-rolling direction.

The only feasible method for studying two-dimensional preferment such as this is to make a pole figure for the specimen. Such a pole figure is a summarization by stereographic projection of data obtained for one set of crystal planes from a series of X-ray photographs taken at different angles to the sheet.[2] It is a record made in such a way that it may be easily interpreted. The pole figure is a circle which is the stereographic projection of an imaginary spherical surface at the center of which a crystal is assumed to be placed. Each point on the sphere is represented by a point on the projection, and each possible orientation of a plane in the crystal is represented by a point on the sphere, located at the intersection with the surface of the sphere of the normal to the plane (perpendicular to the plane) which passes through the center of the sphere. Thus each point on the pole figure corresponds to a definite orientation of the set of crystal planes being studied. The relative number of planes in the sample which are oriented in any one direction is represented by the density of the corresponding spot on the pole figure. For random orientation of grains in a specimen, that is, if every possible orientation exists to the same degree, the pole figure will be of uniform low density. The pole figure of a single crystal will contain regularly placed points of maximum density, the remainder of the figure having zero density. The positions of these points tell the interpreter the orientation of the crystal, because they represent the directions of the normals to the crystal planes concerned. In a rolled metal the crystal orientation is somewhere between that of an ideal crystal and that of random orientation. The pole figure of the rolled specimen shows by its varying density the extent and directions of deviations from the ideal single crystal orientation.

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[1] Physicist, Research Laboratory, General Electric Co., Schenectady, N. Y.

[2] There are other means of obtaining data for a pole figure (for example, by optical reflection from etch pits, X-ray back reflection, etc.), but the X-ray transmission method has widest application because of the ease with which information may be obtained.

It is the purpose of the author in writing this paper to explain a method for making pole figures without giving any discussion of the theory of the stereographic projection[3] or of the spherical geometry and trigonometry on which the construction of pole figures is based. It is hoped that in this way the simplicity of the method will not be disguised.

EXPERIMENTAL SETUP

Preparation of Samples:

The photographs made to obtain data for pole figures require long exposures because of the distances necessary in the camera arrangement. Therefore, it is advantageous to minimize as much as possible any other factors which would cause an increase in exposure time. Since the X-ray beam must pass through the sample (see the section on Nature of Patterns Produced) there will be absorption of the X-rays; thus, the thickness of the sample should be made as small as is mechanically feasible; for example, a thickness of 5 to 6 mils is quite satisfactory for steel. Reduction in thickness must be achieved by methods which will not change in any way the structure of the metal. Obviously, rolling the sample down to the desired thickness is entirely out of the question. Grinding or filing may be used as a reduction method if the operation is done with special care to keep surface flow of metal at a minimum and also if it is followed by an etch to remove those surface effects which do appear because of the grinding or filing. This etch must be done with a reagent which will give a uniform attack on the surface of the metal. The reduction may also be done entirely by etching, but probably a combination of grinding and etching is most satisfactory.

If both surfaces of the specimen are reduced, the sample obtained is the midsection of the specimen. To obtain an outer section for study, only one surface must be reduced. If the reduction is done by etching, one surface of the specimen should be coated with paraffin to protect it from the etching solution.

If the grain size in the rolled metal is uniform and small enough so that the X-ray beam passes through a representative number of grains with the specimen stationary, the sample may be prepared in the shape of a rod rather than a sheet.[4] This eliminates any changed intensity effect due to increased absorption as the inclination of the sample to the beam is decreased (see Section on Nature of Patterns Produced.) Where it is necessary to study extremely thin layers of material, this shape of sample is, of course, not practicable, because of the difficulty in preparing a perfect cylinder without destroying the worked characteristics of the sheet.

The surfaces of the sample should be kept clean at all times so that no confusing effect will be introduced by the presence of oxides.

Camera Arrangement:

The photographs necessary to obtain data for a pole figure are transmission Laue patterns taken with a chosen direction in the sample perpendicular to the X-ray beam, while the plane of the sample is placed at various angles to the beam. A complete series of patterns corresponding to the reflection circles on the pole figure chart is usually taken with the rolling direction perpendicular to the X-ray beam; in addition, a number of patterns is taken with the cross-rolling direction perpendicular to the beam sufficient to give data for the region included in the tangent circles of the pole figure chart. (For description and explanation of the pole figure chart, see section on Pole Figure Chart.)

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[3] For a discussion of the theory, the reader is referred to the article "The Stereographic Projection" by Charles S. Barrett, *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 124, p. 29 (1937).

[4] P. W. Bakarian, "Preferred Orientation in Rolled Magnesium and Magnesium Alloys," *Transactions*, Inst. of Metals Div., Am. Inst. Mining and Metallurgical Engrs., Vol. 147, p. 266 (1942).

For accurate determination of the position of the sample with respect to the beam, the sample holder should be equipped to rotate the sample about the chosen axis without requiring its removal from the holder or removal of the holder from its position. A vernier scale on the holder for reading the angle of rotation will also increase the accuracy of the position determination.

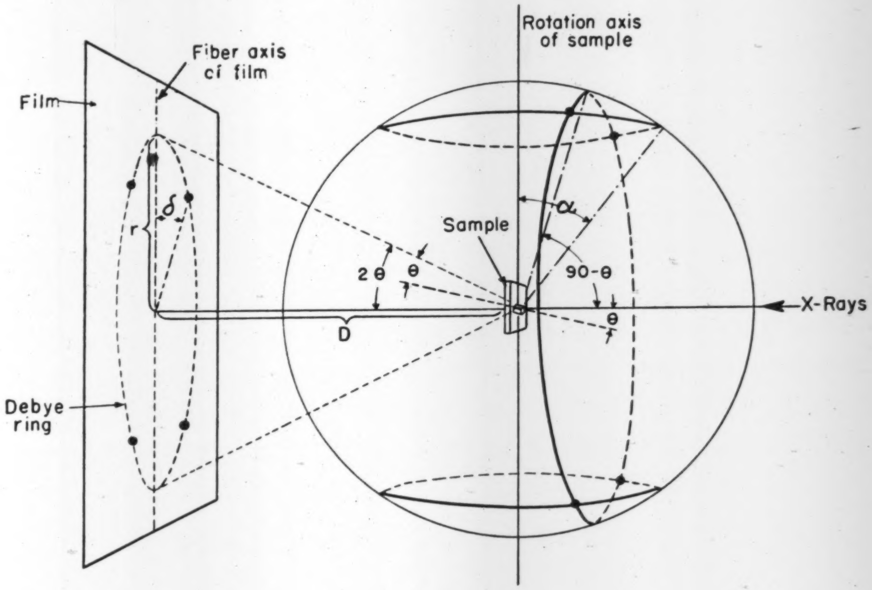

FIG. 1.

If the sample is not sufficiently fine-grained to eliminate the possibility of a spotty pattern, it is necessary that an integrating holder be used so that during the exposure the X-ray beam will scan a large representation of grains, much as the eyes of the reader scan the printed words on these pages.

In choosing distances to be used in the camera setup, the experimenter should consider three points: (*1*) the intensity of the radiation striking the film varies inversely with the square of the distance from the focal point of the X-ray target to the photographic film. Therefore, to keep exposure time low, it is well to make this distance as small as possible. However, (*2*) the ease and accuracy of the measurements is increased if the size of the Debye ring is increased, which is accomplished by making the distance from sample to film greater (see Section on Nature of Patterns Produced). Thus, one must strike a medium between (*1*) and (*2*) in choosing the distance from sample to film. A radius of 3 to 4 cm. for the Debye ring gives fairly accurate pole figure data. (*3*) If the film cassette is made large enough, rings outside of the particular Debye ring chosen for the pole figures will also be recorded on the film. Information obtained from them is often useful afterwards to the investigator. The film cassette may be made circular or rectangular and should be made a standard film size so that there is a minimum of waste in cutting the film. Films 8 by 10 in. may be used to good advantage.

NATURE OF PATTERNS PRODUCED

The X-ray pattern produced on the film will be a series of Debye rings concentric with the undeviated X-ray beam. One such ring is sketched in the schematic diagram shown in Fig. 1. The radius, r, of a Debye ring is given by the formula

$$r = D \tan 2\theta$$

where D is the perpendicular distance from sample to film, and θ is the angle between the X-ray beam incident on the sample and the family of crystal planes which reflect to form the particular Debye circle under consideration. In the figure, one unit cell is sketched in proper position for the upper face to reflect rays to the top of the Debye circle; θ is called the Bragg angle of incidence, and it is given by the formula

$$n\lambda = 2d \sin \theta$$

where:

$$d, \text{the interplanar spacing} = \frac{a_0}{\sqrt{h^2+k^2+l^2}}$$

for the cubic system.

Here λ = the wave length of X-radiation used,
hkl = Millerian indices of the planes,
a_0 = length of edge of one cell of atomic structure of crystal (lattice parameter), and
n = order of diffraction ring.

(Sample calculations of θ and r are given in the Appendix.[4a])

The intensity of the Debye ring (assuming all diffracted rays traverse equal distances through the sample) will range from uniform density for completely random orientation of crystalline grains in the sample to spots of maximum density separated by regions of zero density for the perfect orientation of a single crystal sample. (An ideal four-spot pattern for a single crystal is shown in the diagram of Fig. 1.) For pole figure work one is concerned with a sample having some unknown condition of preferred orientation between that of a single crystal and random orientation. For the unknown sample, the intensities on the Debye ring may vary between the above-mentioned limits; and, regardless of the crystalline orientations present in the sample, the intensity at any one point on the Debye ring is directly proportional to the number of planes which are in position to reflect rays to the point. The position of any plane which causes a reflection to a point on the Debye ring, such as that just mentioned, is given by the cosine law,

$$\cos \delta = \frac{\cos \alpha}{\cos \theta}$$

where:
α = angle between normal to reflecting planes and rotation axis of sample,
δ = angular departure of spot from fiber axis of film (azimuth angle), and
θ = Bragg angle of incidence. (See Fig. 1.)

In Fig. 1, four spots are shown in the imaginary sphere of reference drawn around the sample. These spots are the intersections with the reference sphere of the normals to the planes which cause the four spots on the film, since for each spot on the reference sphere the values of α and θ are the proper values for the corresponding spot on the film.

The Debye circles commonly used for pole figures of metals correspond to very low values of θ, and so $\cos \theta \cong 1$ in the formula given above. Therefore, $\delta = \alpha$, and a measurement of an angle on the film is a direct determination of the angle between corresponding directions in the sample.

[4a] See p. 799.

The X-ray beam passes through the sample before falling on the photographic film and, therefore, the intensity is decreased by absorption within the sample. With sheet samples, except when the X-ray beam is perpendicular to the rolling plane, the absorption is not the same for all points on the Debye ring, since, in passing through the sample, the diffracted rays traverse paths of unequal lengths. Maximum absorption (minimum transmitted intensity) occurs for the beam diffracted to the point on the cross-rolling direction of the Debye ring nearer the portion of the sample inclined toward the film, and minimum absorption occurs for the beam diffracted to the opposite point of the Debye ring. The ratio of minimum transmitted intensity to maximum decreases as the angle, γ, between the X-ray beam and the normal to the surface of the sample increases.

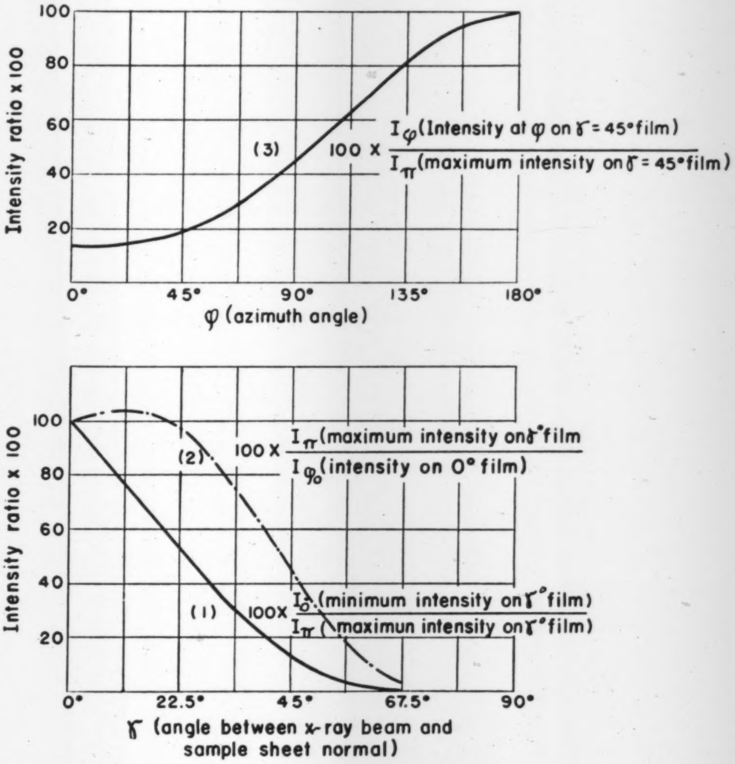

FIG. 2.—Intensity Variations Caused By Absorption.
Curves drawn for the first order (110) ring for silicon steel 5 mils thick, using molybdenum Kα radiation.

Of course, the variation in intensity considered here is that for random orientation in the sample, and any variation due to preferred orientation is superimposed on this.

The intensity I_ϕ, transmitted to a point on the Debye ring with azimuth angle, ϕ, as measured from the cross-rolling direction on the side nearer to the sample, may be expressed:

$$I_\phi = I_0 \frac{A}{B}(e^{Bt} - 1)$$

where:

$A = e^{-\mu t C}$,

$B = -\mu\left(\frac{1}{\cos\gamma} - C\right)$,

$C = \frac{1}{\cos\gamma\cos 2\theta - \sin\gamma\sin 2\theta\cos\phi}$,

μ = coefficient of absorption,
t = thickness of sample,
γ = angle between the X-ray beam and the normal to the surface of the sample, and
θ = Bragg angle of incidence.

In Fig. 2, three curves are shown to illustrate the intensity variations caused by absorption. They are drawn for the first order (110) ring for silicon steel 5 mils thick, using molybdenum K_α radiation. Curve 1 shows how the ratio of minimum to maximum intensity varies with γ. This ratio decreases quite rapidly, and for the higher angles considerable error is introduced if the absorption variation is neglected. The ratio of the maximum intensity to the intensity obtained with the sample surface normal to the beam varies with γ as shown in curve 2. This quantity has a maximum at $\gamma = \theta$ (10.12 deg.), and decreases with continued increase in γ. Curve 3 shows the variation of the ratio of intensity at azimuth 0 to maximum intensity for the $\gamma = 45$ deg. film.

It should be remembered that the intensities considered in these calculations are actual transmitted intensities, and the large differences will not be so marked on the photographic film because of its blackening limit. It is suggested that the exposure times of the photographs be varied so that the maximum intensities will be the same for each film. This will eliminate some of the discrepancies caused by the absorption. In addition, for the higher angle films it would be well to use only the more intense half of the Debye ring, which means using only half of each corresponding reflection circle on the pole figure.

If data are obtained by some method other than photographing, more exact corrections, using the formula above, will be necessary.

(Derivation of the intensity formula, sample calculations and a "pole figure" for random orientation are given in the Appendix.)

POLE FIGURE CHART

In pole figure work it is usually desired to make a comparison for a series of specimens, and for this purpose a number of pole figures must be made for the same value of θ, that is, the same crystal plane, lattice constant, and radiation. (See Appendix for sample calculation of θ.) The labor of this process is greatly decreased if a pole figure chart is made for the given values of θ. This may be done in the following way:

Choose a standard stereographic or Wulff net of the size desired for the pole figure, since the periphery or boundary circle of the Wulff net will also be the boundary circle of the pole figure. Place a piece of transparent tracing material, for example, lumerith, over the Wulff net, and trace onto it the equator, vertical meridian and boundary circle of the net.

Data obtained from the proper Debye ring on the 0 deg. film (film taken with the specimen at right angles to the X-ray beam) will be recorded on a circle, known as the 0 deg. reflection circle, which, by the theory of stereographic projection, must be concentric with the boundary circle of the pole figure. On the pole figure chart, draw this 0 deg. circle with radius $90 - \theta$ deg. as measured on the equator, or the vertical meridian of the Wulff net.

To determine the parallels of latitude for the pole figure chart, divide each quadrant of the 0 deg. circle just constructed into 10-deg. arcs by drawing radial lines at 10-deg. intervals and extending them to the boundary circle. (These should be erased after the chart is completed.) The intersections of the radial lines with the 0 deg. circle are the points where the 10 deg. parallels of latitude for the pole figure chart cut the 0 deg. circle. Draw in the parallels of latitude, complete to the boundary circle by tracing them from those of the Wulff net. If they fall between two latitude lines of the net, use visual interpolation in making the traces. If parallels of latitude are desired at closer intervals, they may be made in the same way.

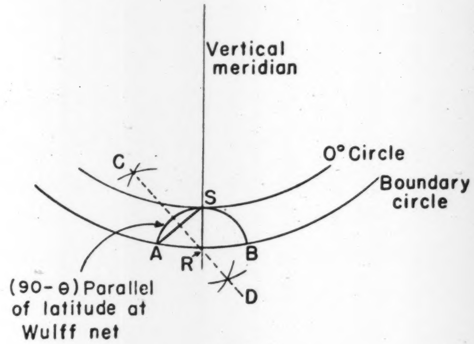

FIG. 3.

At the north and south poles of the 0 deg. circle, construct the tangent circles which contain the $90 - \theta$ deg. parallels of latitude of the Wulff net. The following is a suggested method: By the rules of stereographic projection, the center of the tangent circle must be on the extension of the vertical meridian. To locate it draw a line (*SA* in Fig. 3) from the pole, *S*, of the 0 deg. circle to the intersection, *A*, of the $90 - \theta$ deg. parallel of latitude of the Wulff net with the boundary circle. This line will be a chord of the tangent circle, and so its perpendicular bisector must pass through the center of the circle. Therefore, the point, *R*, where the perpendicular bisector, *CD*, intersects the vertical meridian will be the center of the tangent circle. The location of *R* should be checked using a similar treatment with *B* instead of *A*. When *R* has been determined, draw the tangent circle with radius *RA*.

The 0 deg. circle, as stated above, is the reflection circle to be used when the sample is at right angles to the X-ray beam. For this case the plane of the Debye ring on the film is parallel to the plane of the sample. Now suppose the sample is rotated through *n* degrees about an axis represented by the vertical meridian of the pole figure chart (usually the rolling direction for rolled sheets). The Debye ring is, of course, still circular, but it is no longer parallel to the sample. However, because of the fact that the stereographic projection of a circle must always be a circle, the data from this *n* deg. film will be recorded on circles; or, in the event that the inclination of sample to film is great enough to throw a portion of the circle outside of the boundary circle, the data will be recorded on circular arcs, one circular arc being the portion within the boundary circle, the other the reflection of the portion which lies outside of the boundary. Specifically, data will be recorded on two circles or four circular arcs. These may be determined by the following method:

The centers of the circles, by the rules of stereographic projection, must lie on the equator (or equator extended).

One circle will intersect the equator at points located *n* deg. to the right of the 0 deg. circle, as measured on the Wulff net. In the right half of the chart the number of degrees on the equator of the Wulff net to the right of the 0 deg. circle may be less than *n* deg. in which case the desired point is found by retracing a path along the equator from the boundary circle to the left for a distance in degrees equal to the difference between *n* deg. and the number of degrees on the equator of the Wulff net between the 0 deg. circle and the boundary circle. For example, suppose $n = 30$ deg. and the number of degrees between the 0 deg. circle and the boundary circle, as read on the equator of the Wulff net, is 11 deg.; then the point desired will be found by retracing 30 − 11 deg. = 19 deg. to the left from the boundary circle, also as read on the equator of the Wulff net. When it is necessary to retrace a path in order to locate the intersecting point, the projection sought will be two circular arcs instead of one complete circle.

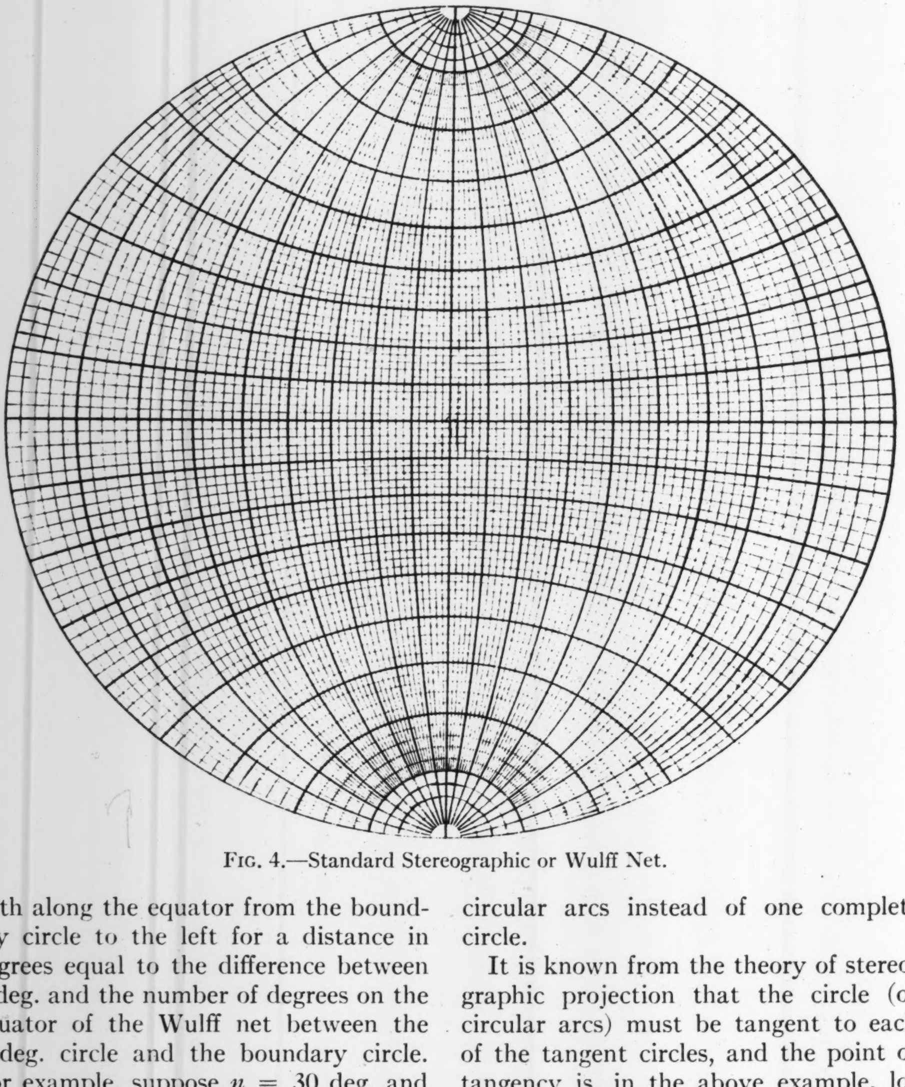

FIG. 4.—Standard Stereographic or Wulff Net.

It is known from the theory of stereographic projection that the circle (or circular arcs) must be tangent to each of the tangent circles, and the point of tangency is, in the above example, located at a point *n* deg. to the right of the vertical meridian, measured on the periphery of the tangent circle by the intersection of an *n* deg. radial line of that circle. When these points are located, the center of the *n* deg. circle can be determined in the same way that the centers of the tangent circles were determined, or it may be determined with sufficient accuracy for most pole figure work by a simple trial-and-error method. In the case of two circular arcs, the left arc should be constructed first, and the intersection it makes with the boundary circle can be used, together with the proper point on the equator, to determine the center of the right arc. The other *n* deg. reflection circle is located in a similar way to the left of the 0 deg. circle.

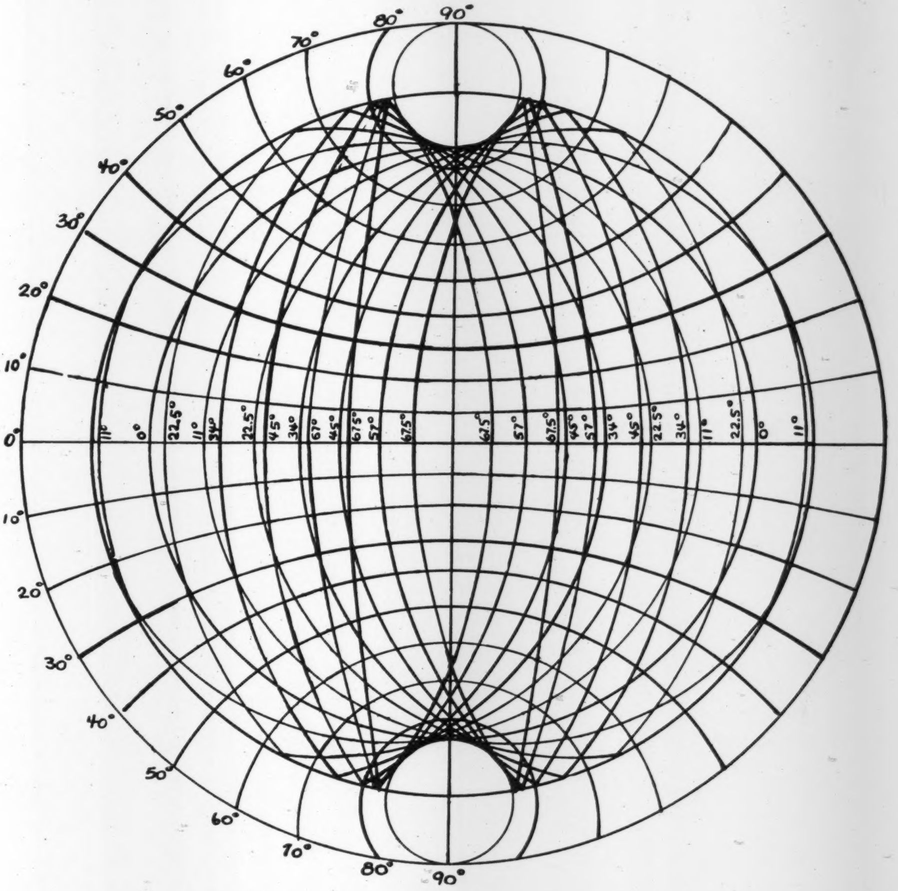

FIG. 5.—Pole Figure Chart for (110) Planes of Silicon Steel, $a_0 = 2.9566\text{Å}$, and Molybdenum K_α Radiation.

The pole figure chart may be made with the reflection circles at intervals of 5 to 15 deg., depending on the accuracy desired.

Fig. 4 shows a standard stereographic or Wulff net[5]. Figure 5 shows a pole figure chart which has been constructed over a Wulff net and the Wulff net removed [(110) planes of silicon steel, $a_0 = 2.9566$ Å; molybdenum K_α radiation].

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[5] This is a photographic reproduction of a Wulff net and should not be used for making a pole figure chart. Accurate Wulff nets may be procured from the United States Hydrographic Office.

POLE FIGURE

After the pole figure chart has been made for the proper value of θ, the necessary number of diffraction patterns of the sample taken, and the desired Debye rings identified on the films (see sections on Experimental Setup, Nature of Patterns Produced, and Pole Figure Chart), the experimenter is ready to construct the pole figure itself. Necessary data may be taken from the film in the following way:

Determine the intensity distribution around the Debye circle by marking off each maximum into zones of equal intensity. This may be done with the aid of a microphotometer trace, or the zones may be estimated visually by using a polar net (see Fig. 6). In the latter case, the author has found that four- or five-zone rating (going from zero to maximum intensity) may be used to good advantage. Choose some reference point on the Debye circle (preferably the cross-rolling direction) and record the zone

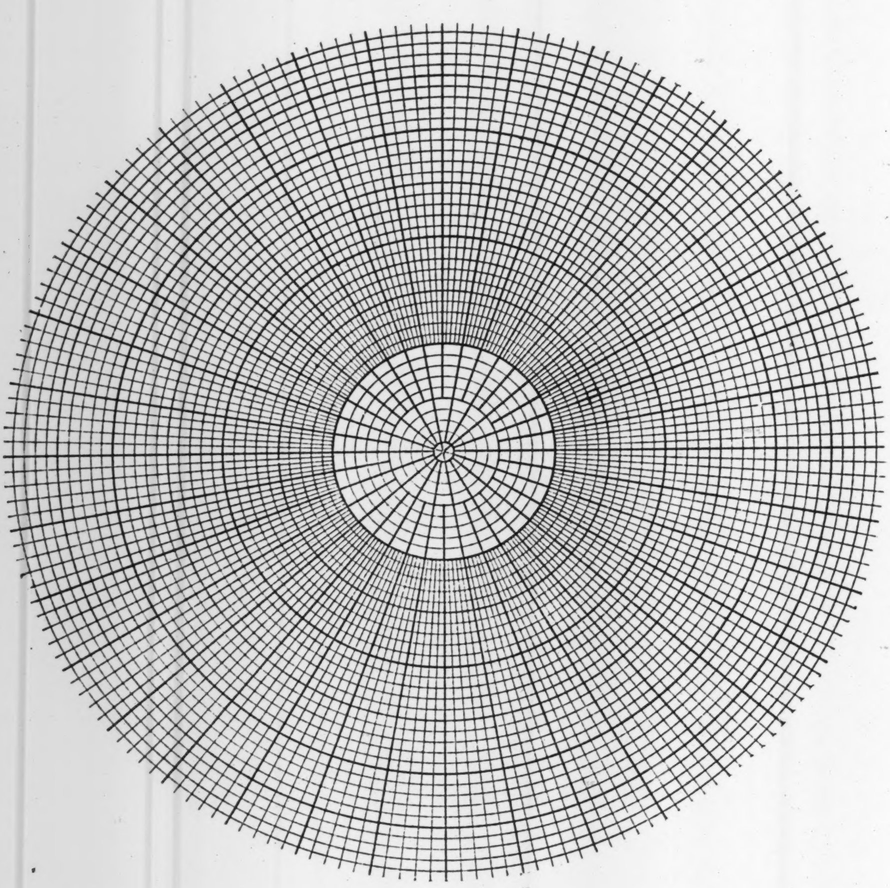

FIG. 6.—Polar Net.

boundaries in degrees with respect to this reference point. Similar data must be taken for each film. An example is shown in Fig. 7. The sample is a cold-rolled silicon steel strip, and this photograph is taken with the plane of the sample at an angle of 34 deg. with respect to the X-ray beam. The pole figure is to be made for the (110) planes, and since these are the planes of greatest atomic density, data are taken from the white radiation ring—the wide, innermost ring. The 110 Debye circle for the characteristic radiation lies just outside the white radiation ring; note that the intensities on this circle vary in exactly the same manner as those caused by the white radiation. The intensities are designated 0, 1, 2, 3, that is, in terms of a four-zone rating, where 0 indicates zero intensity and 3 indicates maximum intensity. (No absorption corrections

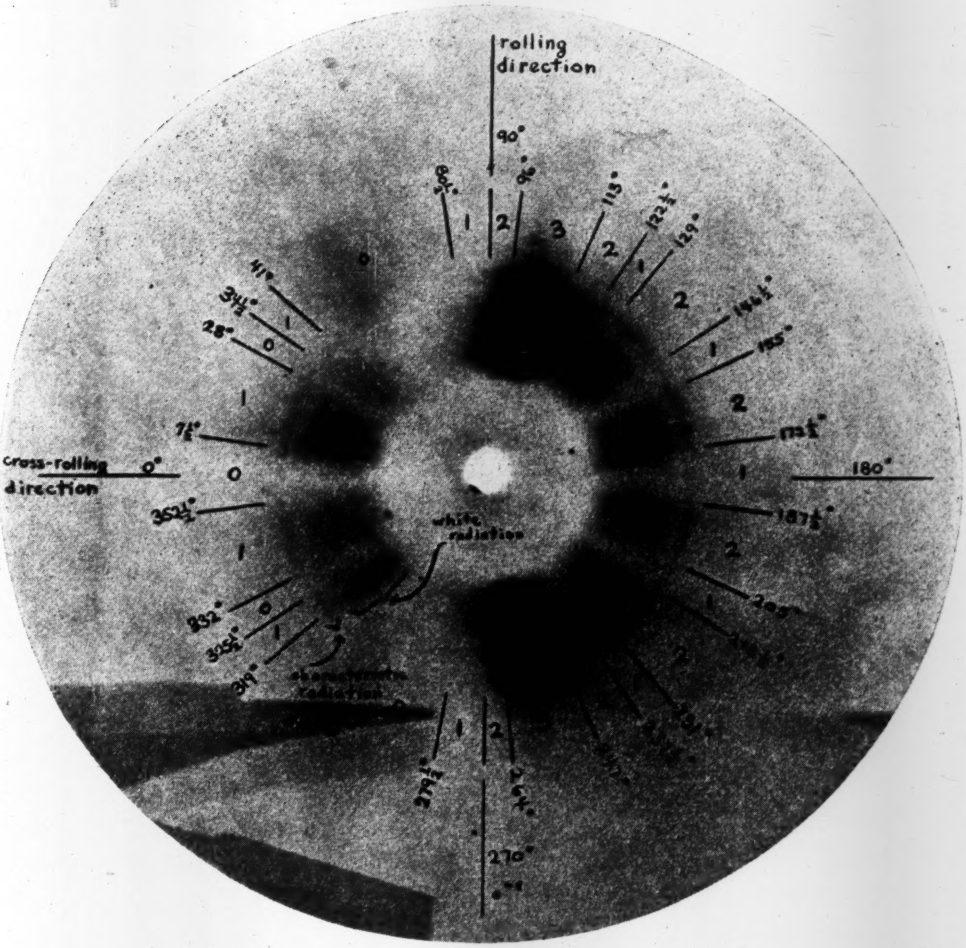
rolling direction
90°
80½°
96°
1
2
3
113°
2
122½°
1
129°
2
146½°
1
155°
2
173½°
1
180°
187½°
2
205°
1
2
231°
2
264°
270°
279½°
1
cross-rolling direction
0°
0
41°
34½°
1
28°
0
0
1
7½°
352½°
1
332°
0
325½°
1
319°
white radiation
characteristic radiation

FIG. 7.—Transmission X-ray Photograph of a Cold-Rolled Silicon Steel Strip, Taken with the Plane of the Sample at an Angle of 34 deg. with Respect To the X-ray Beam.
Intensity variations are marked on a scale of 0 to 3.

are made for variation around the Debye circle on this film.)

To make the pole figure, place a sheet of tracing cloth over the pole figure chart, trace the boundary circle, and mark the rolling and cross-rolling direction. The data taken from each film in the manner described above must be recorded on the proper reflection circles of the pole figure chart. The method of recording may best be explained by an example. Let us consider the silicon steel 34 deg. film measured above. Data from this film must be recorded on the 34 deg. reflection circles (see Fig. 8). A scale of differentiation must be chosen, and the author has found the scale given in the figure to be quite satisfactory. The cross-rolling direction was taken as the direction of reference on the film; therefore, data are recorded with respect to the cross-rolling direction of the pole figure. On the film the reference cross-rolling direction is marked 0 deg. This corresponds to the point *A* on the cross-rolling direction of the pole figure. Note that on the film there is a region of 0 intensity for $7\frac{1}{2}$ deg. either side of 0 deg.; therefore, for $7\frac{1}{2}$ deg. either side of *A* on the 34 deg. circle of the pole figure, there is a region of 0 intensity, which is left blank according to the scale of differentiation. The boundaries of this 0 intensity zone on the film are also boundaries of 1 intensity zones whose other boundaries are at 28 deg. on either side of 0 deg. On the pole figure these boundaries lie 28 deg. either side of *A*.

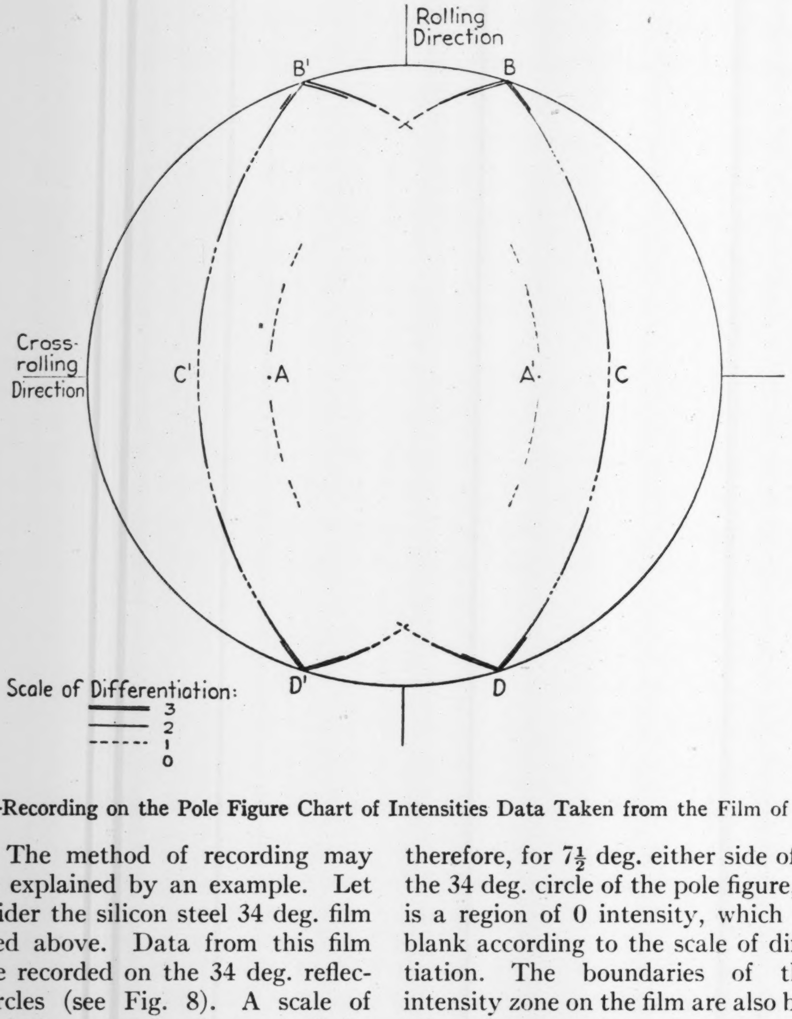

FIG. 8.—Recording on the Pole Figure Chart of Intensities Data Taken from the Film of Fig. 7.

The regions between the boundaries of these 1 intensity zones on the pole figure are marked with dashed lines as designated for intensity 1 on the scale of differentiation. These regions are bounded by 0 intensity zones to 41 deg. either side of *A*, etc. Starting from *A′* instead of *A*, the same data are recorded around the arcs *A′B′C′D′A′*. The arcs designated by primed letters are reflections in the rolling axis of those designated by unprimed letters, and they correspond to a film which would be taken with the sample at an angle of 34 deg. to the X-ray beam but in a direction opposite to that for which the film shown above is taken. Data from each film are to be recorded in the same manner on the corresponding reflection circles.

On the 0 deg. film, that is, film with the sample perpendicular to the X-ray beam, the pattern is symmetrical with respect to the rolling and cross-rolling directions, but as the sample is rotated around the rolling direction the pattern becomes more and more unsymmetrical with respect to the rolling direction although it retains symmetry to the cross-rolling direction. This accounts for the large degree of asymmetry present on the 34-deg. film shown above.

When all data have been recorded on the pole figure, join like zone boundaries with smooth curves. The pole figure will thus be divided into zones of different intensity. Visualization and subsequent interpretation will be made easier if differentiation between zones is accomplished by filling in the zones with different colors, cross-hatching, or different degrees of shading.

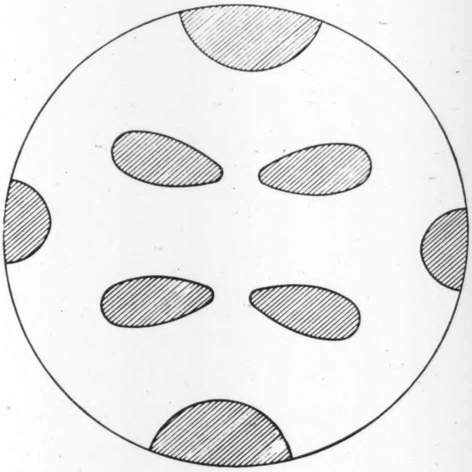

FIG. 9.—(110) Pole Figure for the Midsection of a Strip of Cold-Rolled Silicon Steel Reduced from 0.108 in. in Thickness to 0.038 in. in Thickness (64.8 per cent).

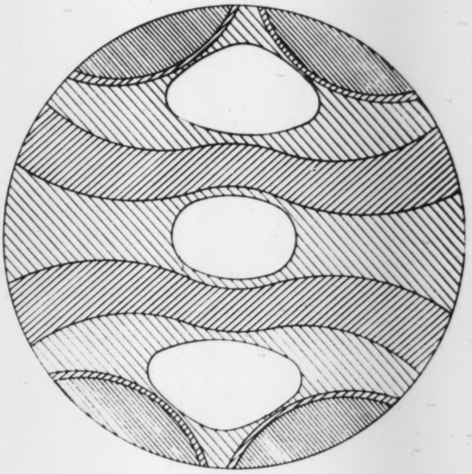

FIG. 10.—Pole Figure for the Outer Section of the Sample of Silicon Steel Used for Fig. 9.

The pole figure is now completed and ready for interpretation.

Some examples of pole figures are shown in Figs. 9 and 10.

INTERPRETATION OF POLE FIGURES

A general scheme for interpretation of pole figures will be explained, followed by special examples.

Assume the rolling direction of the specimen to correspond to the north-south direction of the pole figure; the equator will be the cross-rolling direction. The orientations of the normals to the planes under consideration which give rise to the various spots on the pole figure may be found as follows: Determine the colatitude, n, of the spot, and the meridian angle, m (where $m \leq 90$ deg.), between the spot and the boundary circle of the pole figure. The normal to the planes responsible for this spot will make an angle n to the rolling direction of the sample, and the plane determined by this normal and the rolling direction will make an angle m with the plane of the specimen.

From this general rule, the following rules for specific points may be written down:

A.—Central point of the pole figure—the normal to the crystal planes is perpendicular to the plane of the specimen. (The plane of the sample and the reflecting plane are coincident.)

B.—Points on the circumference of the pole figure—the normal to the crystal planes lies in the plane of the specimen. (The reflecting plane is perpendicular to the plane of the sample.)

a.—Points at the north and south poles—the normal lies along the rolling direction. (The reflecting plane is perpendicular to the rolling direction.)

b.—Points at the equator—the normal lies along the cross-rolling direction. (The reflecting plane is perpendicular to the cross-rolling direction.)

c.—Points with co-latitude n deg.—the normal makes an angle n deg. with the rolling direction. (The reflecting plane makes an angle $90 - n$ deg. with the rolling direction.)

C.—Points on the equator m deg. from the boundary circle—the normal to the crystal planes is perpendicular to the rolling direction, and makes an angle m deg. to the cross-rolling direction. (The reflecting plane makes an angle $90 - m$ deg. to the cross-rolling direction.)

D.—Points on the vertical meridian with co-latitude n deg.—the normal to the crystal planes is perpendicular to the cross-rolling direction and makes an angle m deg. to the rolling direction. (The reflecting plane makes an angle $90 - n$ deg. to the rolling direction.)

Often it is possible to determine the crystal orientation shown on the pole figure by a simple trial-and-error method, using a model of the crystalline unit cell. The model is placed at the center of the pole figure in various positions until one is found for which the normals of the planes under consideration are in proper position to give the pattern of the pole figure.

Figure 9 shows a (110) pole figure for the mid-section of a strip of cold-rolled silicon steel reduced from 0.108 in. in thickness to 0.038 in. in thickness (64.8 per cent). This is close to an ideal orientation with a [110] direction in the rolling direction and a (100) plane in the plane of the sample. Of course, for completely ideal orientation the spots on the pole figure would be points only. Thus, by measuring the dimensions of the spots, in degrees, on the actual pole figure, one has a measure of the deviation from ideal orientation. In the sample which Fig. 9 represents, the orientation of the crystal grains varies within a range of ±10 to 15 degrees in all directions from the ideal position.

Figure 10 is a pole figure for the outer section of the sample of silicon steel used for Fig. 9. It shows an orientation much farther from the ideal case than that of Fig. 9. The [110] directions, instead of being concentrated about the rolling direction as in Fig. 9 are concentrated about two directions approximately 25 deg. either side of the rolling direction. Also a much greater range of deviations in directions perpendicular to the rolling direction is indicated by the two less concentrated bands of intensity maxima which spread across the pole figure.

The two pole figures just considered are extremes. For most specimens of cold-rolled silicon steel, the pole figure will show a structure somewhere between those of Figs. 9 and 10.

Acknowledgments:

The author wishes to express her appreciation to L. L. Wyman[6] and David Harker of the General Electric Co. for their comments and constructive criticism.

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[6] Loaned to the War Metallurgy Committee of the National Research Council.

APPENDIX

Sample calculations of θ and r for the 1st order (110) ring for silicon steel, using molybdenum K_α radiation, and with the sample at a distance of 10 cm. from the film:

known data: $n = 1$
$hkl = 110$
$a_0 = 2.8566 \text{ Å} = 2.8566 \times 10^{-8}$ cm.
$\lambda = 0.710 \text{ Å} = 0.710 \times 10^{-8}$ cm.
$D = 10$ cm.

$$\sin \theta = \frac{n\lambda \sqrt{h^2 + k^2 + l^2}}{2a_0} \ldots \text{(see p. 788)}$$

$$\sin \theta = \frac{1 \times 0.710 \times \sqrt{2}}{2 \times 2.8566} = 0.1757$$

$$\theta = 10.12^\circ$$

$$r = D \tan 2\theta \ldots\ldots\ldots\ldots\ldots \text{(see p. 788)}$$

$$r = 10 \times 0.3687$$

$$r = 3.687 \text{ cm.}$$

ABSORPTION FORMULA

Derivation:

Figure 11(*a*) shows a portion of a rolled metal sheet with an X-ray beam diffracted in passing through it. The sample of thickness t, makes an angle γ with the plane normal to the X-ray beam. Consider the ray *ABEH* which is diffracted at B through the angle 2θ and strikes the film at H, the film being perpendicular to the direction of the original X-ray beam. The azimuth angle *HGM*, where *GM* is the cross-rolling direction on the film is denoted by ϕ. C is the intersection with the sample surface of the direct X-ray beam, and E is the point where the ray under consideration intersects the surface. Figure 11(*b*) shows the Debye circle through E. The direct X-ray beam is perpendicular to the plane of the figure and passes through D; therefore, the portion of this Debye circle above the horizontal line through E lies inside the sample, the lower portion lies outside, and the plane of the circle makes the angle γ with the sample (that is, $< DFC = \gamma$). *ED* and *FD* in Fig. 11(*b*) are parallel to *HG* and *MG*, respectively, in Fig. 11(*a*). Thus, $\angle EDF = \varphi$.

Let d be the total distance traversed by the diffracted ray in passing through the sample. Then

$$d = AB + BE \ldots\ldots\ldots (1)$$

$$AB = \frac{x}{\cos \gamma} \ldots\ldots\ldots\ldots\ldots (2)$$

$$BE = \frac{BC + CD}{\cos 2\theta} \ldots\ldots\ldots (3)$$

since *FD* is perpendicular to the direct beam *ABCDG*. Also

$$BC = \frac{t - x}{\cos \gamma} \ldots\ldots\ldots\ldots (4)$$

and

$$CD = FD \tan \gamma = ED \cos \varphi \tan \gamma$$

But ED is perpendicular to BCD, so that ED = BE sin2θ. Therefore,

$$CD = BE \sin 2\theta \cos \varphi \tan \gamma \quad \ldots\ldots (5)$$

Combining Eqs. 3, 4, and 5

$$BE = \frac{\dfrac{t - x}{\cos \gamma} + BE \sin 2\theta \cos \varphi \tan \gamma}{\cos 2\theta}$$

or

$$BE = \frac{t - x}{\cos \gamma \cos 2\theta - \sin \gamma \sin 2\theta \cos \varphi} \quad \ldots (6)$$

and from Eqs. 1, 2 and 6

$$d = \frac{x}{\cos \gamma} + \frac{t - x}{\cos \gamma \cos 2\theta - \sin \gamma \sin 2\theta \cos \varphi} \quad \ldots (7)$$

The intensity at H due to ray $ABEH$ will be

$$I_\varphi(x) = I_0 e^{-\mu d} \quad \ldots\ldots\ldots\ldots (8)$$

where:

μ = the coefficient of absorption of the material, and

I_0 = a constant depending on the initial intensity of the X-ray beam and the fraction of the original beam that is diffracted.

To get the total intensity at H, this function must be integrated from $x = 0$ to $x = t$, since rays are diffracted to H from all points along AC. Thus,

$$I_\varphi = I_0 A \int_0^t e^{Bx} dx = I_0 \frac{A}{B} (e^{Bt} - 1) \quad \ldots (9)$$

where:

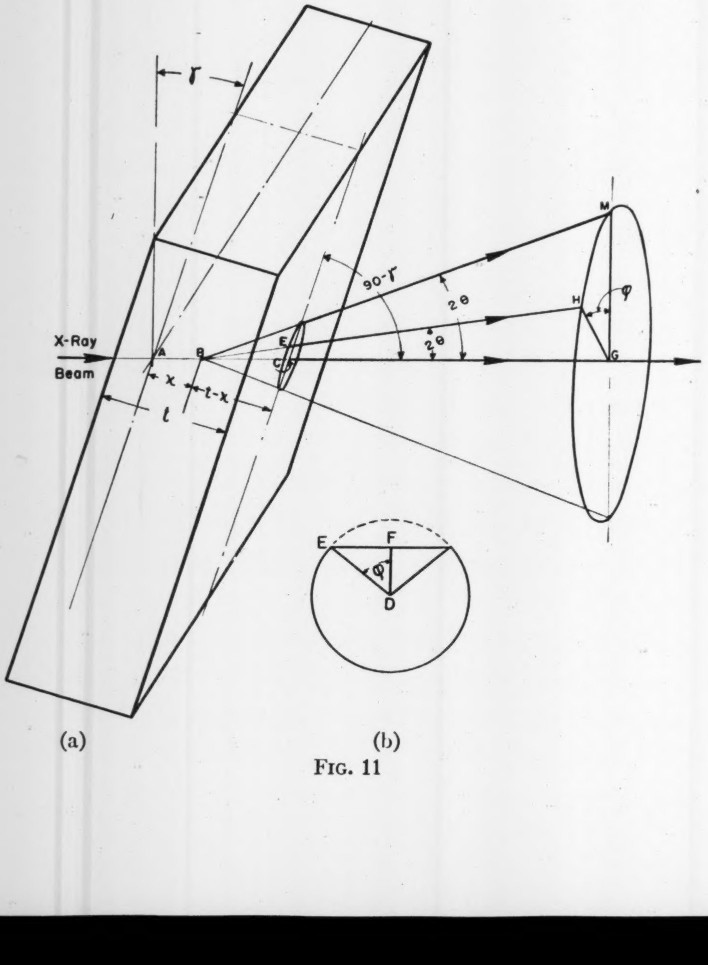
$$A = e^{-\mu t C}$$

$$B = -\mu \left(\frac{1}{\cos \gamma} - C \right)$$

$$C = \frac{1}{\cos \gamma \cos 2\theta - \sin \gamma \sin 2\theta \cos \varphi}$$

and $C_{\varphi=0} = 1/\cos (\gamma + 2\theta)$

$C_{\varphi=\pi} = 1/\cos (\gamma - 2\theta)$



(a) (b)

FIG. 11

Sample Calculations: (Silicon steel, 0.005 in. thick, molybdenum K_α radiation)
Known data:

$$2\theta = 20.24°,\ \sin 2\theta = 0.349,\ \cos 2\vartheta = 0.937$$

$$\mu = 302.57$$

$$t = 0.0127\ \text{cm.} \qquad \mu t = 3.843$$

Let $\gamma = \pi/4$ and $\varphi = \pi/3$.

$$C_{\pi/3} = \frac{1}{0.937 \cos \pi/4 - 0.349 \sin \pi/4 \cos \pi/3} = 1.857$$

$$A_{\pi/3} = e^{-3.843 C_{\pi/3}} = e^{-7.14}$$

$$B_{\pi/3} = -302.57 \left(\frac{1}{\cos \pi/4} - C_{\pi/3}\right) = 134.1$$

$$B_{\pi/3}t = 1.703$$

Now let $\varphi = \pi$

$$C_\pi = \frac{1}{\cos (\pi/4 - 20.24°)} = 1.102$$

$$A_\pi = e^{-3.843 C_\pi} = e^{-4.24}$$

$$B_\pi = -302.57 \left(\frac{1}{\cos \pi/4} - C_\pi\right) = -94.5$$

$$B_\pi t = -1.200$$

$$100 \times \frac{I_{\pi/3}}{I_\pi} = \frac{A_{\pi/3} B_\pi (e^{B_{\pi/3}t} - 1)}{A_\pi B_{\pi/3} (e^{B_\pi t} - 1)}$$

$$= \frac{e^{-7.14} (-94.5)(e^{1.703} - 1)}{e^{-4.24} (134.1)(e^{-1.200} - 1)} = 25.0$$

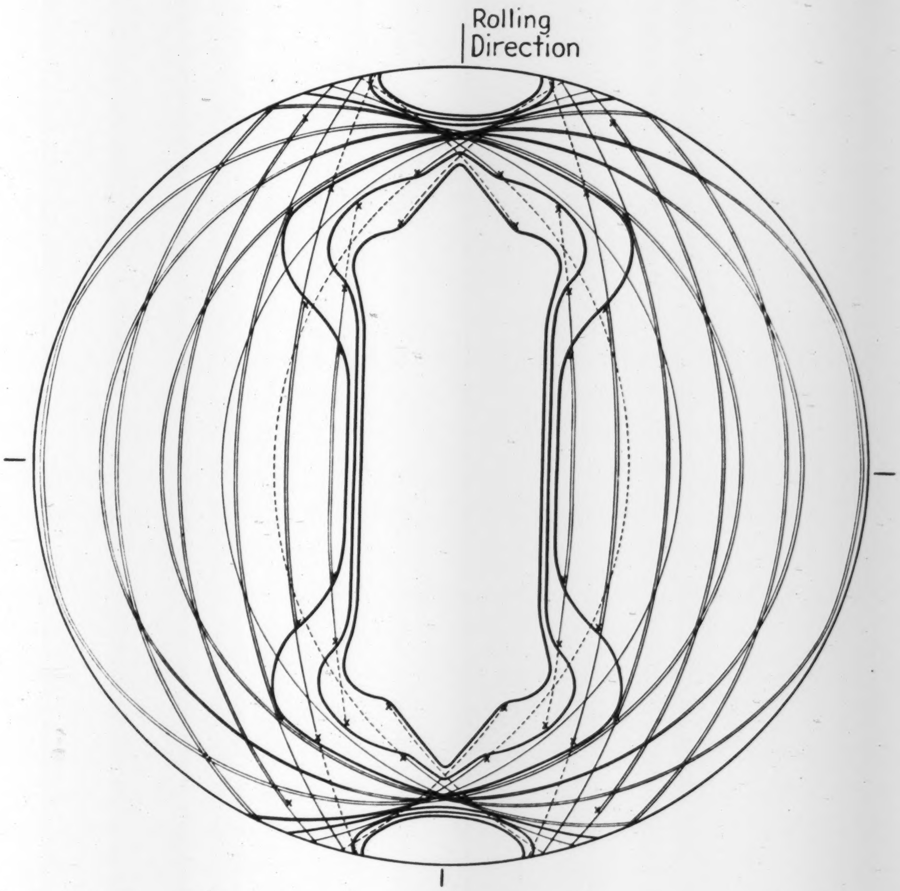


FIG. 12.—"Pole Figure" for Random Orientation, Showing the Effect of Absorption of the X-rays in Passing Through the Specimen.

"Pole Figure" for Random Orientation:

In order to present a clear picture of the effect of absorption variation on the pole figure, curves of type 3, Fig. 2 were constructed for γ = 0 deg., 11 deg., 22.5 deg., 34 deg., 45 deg., 57 deg., 67.5 deg. for 0.005 in. thick silicon steel and molybdenum K_α radiation. Four zones of intensity for each value of γ were marked on the proper reflection circle of the pole figure chart to give the "pole figure" for random orientation, shown in Fig. 12. (This includes the assumption that exposure time is varied properly to make the maximum intensity the same for all values of γ.) The double lines indicate an intensity 75 to 100 per cent of maximum intensity, the single lines 50 to 75 per cent, the dotted lines 25 to 50 per cent, and below 25 per cent the region is left blank. Boundaries of each region are drawn as single heavy black lines, and in sections where two intensities are indicated, only the stronger one is considered in deciding to which region the section belongs; this increases the size of the higher intensity region, thus reducing the error caused by the absorption. The outstanding feature of this figure is the presence of the comparatively large region along the rolling direction in which the intensity is less than 25 per cent of the maximum and consequently practically no information about this region can be obtained from a pole figure as usually made.

SYMPOSIUM ON THE SIGNIFICANCE OF THE HARDNESS TEST OF METALS IN RELATION TO DESIGN

SOME NOTES ON THE INDENTATION HARDNESS TEST

By J. M. Lessells[1]

It is generally accepted that the term hardness has an extremely vague meaning since this may be used to designate a number of quite distinct material characteristics. The expressions "indentation hardness," "cutting hardness," or "abrasion hardness" are frequently used but each covers quite distinct and separate properties. When the engineer uses the term hardness it is generally understood that reference is being made to "indentation hardness."

This Symposium on Hardness, to which this paper is an introduction, was planned as a second of a series, of which the Symposium on Tension Testing was the first,[2] so as to bring together the viewpoint of the physicist, metallurgist and engineer based on present knowledge. These symposiums were planned so as to interpret to the designer the results of present testing practices and the present one on hardness naturally follows that on the tension test.

This symposium, to which this paper is an introduction, brings together the viewpoint of the physicist, metallurgist, and engineer based on present knowledge of hardness. It contains, in addition to this introductory paper, papers by S. R. Williams and S. L. Hoyt, together with the discussion of all three papers. Since frequent use is made of tables to convert from hardness values obtained on one type of machine to those obtained on other types, attention is called to the conversion tables for steels prepared by the Society's Committee E-1 on Methods of Testing.[2a] Moreover, while the introductory paper refers specifically to the Brinell type of test, additional conversion tables form an appendix to that paper. Inclusion has also been made of a selected bibliography covering the more important publications on the subject.

INDENTATION TEST

General:

The indentation test,[3] as is well known, is merely a relative test since the stresses caused by the indentation are unknown and are likely to remain so. The consequences of these stresses can, however, be interpreted, and this has been of great value in the control of materials. In this respect the test possibly occupies a similar place to that of the impact test as being arbitrary but nevertheless extremely useful.

As developed by Brinell[4] the indentation hardness, H, is defined as the unit pressure produced by a load of 3000 kg. through a ball of 10-mm. diameter, the pressure being based on the spherical area rather than the projected area of the resulting indentation. Thus if

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[1] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[2] Symposium on Significance of the Tension Test of Metals in Relation to Design, *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 501 (1940).

[2a] Tentative Hardness Conversion Tables for Steel (Relationship Between Diamond Pyramid Hardness, Rockwell Hardness, and Brinell Hardness) (E 48-43 T), 1943 Supplement to Book of Standards, Part I, p. 342.

[3] Note.—Reference is made in this paper to the Brinell indentation test. The same limitations will apply to other forms of indentation tests.

[4] J. A. Brinell, II^{me} Congrès International des Méthodes d'Essai, Paris (1900).

H = indentation hardness or Brinell number,
P = load in kilograms,
D = diameter of ball in millimeters,
d = diameter of impression in millimeters, and

$$H = \frac{P}{\frac{\pi D}{2}\left(D - \sqrt{D^2 - d^2}\right)}$$

Importance of Geometrical Similarity:

It was appreciated by Brinell that the use of the spherical area in the calculation of the hardness put considerable restriction on its use for loads other than 3000 kg. It can be shown that in order to retain geometrical similarity in the indentations the total angles Φ subtended by the center of the ball and edge of the indentation must be equal for different indentations or

$$\sin\frac{\Phi}{2} = \frac{d}{D}$$

Meyer[5] showed that for a given angle of indentation the mean pressure per unit area is constant irrespective of the ball diameter or

$$\frac{4P}{\pi d^2} = K$$

It therefore follows that for any variation in the ball diameter, so as to allow lighter loads to be used on thin material while retaining similarity, the following relation must hold.

$$\frac{P}{D^2} = K_1$$

Exclusive of manganese steels this allows considerable extension in the use of the indentation hardness test.

Using this relationship some results obtained by Baker[6] are given in Table I.

While the results shown in Table I are fairly uniform for different thicknesses of the same material, further work indicated that when small balls are used the results on the same material may be irregular due to small local differences in the hardness of the material or to a "skin" of different hardness than that of the core.

TABLE I.—COMPARISON OF BRINELL HARDNESS NUMBERS FOR BALLS OF DIFFERENT DIAMETERS.

Steel	Diameter of Ball, mm.	Diameter of Indentation, mm.	Load, kg.	Brinell Hardness Number
A............	10	6.3	3 000	85
A............	7	4.4	1 470	85
A............	5	3.13	750	87
A............	1.19	0.748	42.5	86
B............	10	3.48	3 000	306
B............	7	2.43	1 470	308
B............	5	1.75	750	302
B............	1.19	0.411	42.5	311

Relation Between Indentation Hardness and Other Mechanical Properties:

One of the chief values of the indentation hardness is its relationship to the tensile strength of the material. Greaves[7] showed that if

S = tensile strength in pounds per square inch,[8] and
H = indentation hardness or Brinell number

$$\frac{S}{H} = C$$

where C varies between 470 and 520.

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[5] E. Meyer, Untersuchungen über Härteprufung und Härte, *Zeitschrift des Vereines deutscher Ingenieure* p. 645, (1908).

[6] T. Baker and T. F. Russell, *Journal*, Iron and Steel Inst. (1920).

[7] R. Greaves and J. A. Jones, "The Ratio of Tensile Strength to Brinell Hardness Number," *Journal*, Iron and Steel Inst., Vol. CXIII, p. 335 (1926).

[8] The values given by Greaves for C varies between 0.21 and 0.23 based on S tons per square inch.

A few values for C for different steels based on tensile strength and hardness data given by Hoyt[9] are shown in Table II.

In so far as this relationship refers to quenched-and-tempered steels the Society of Automotive Engineers[10] consider that for carbon and alloy steels the ratio of $\frac{S}{H}$ can be taken as 500 for all practical purposes.

TABLE II.—RELATION BETWEEN TENSILE AND BRINELL HARDNESS NUMBER FOR STEELS.

S.A.E. Steel	Grade	State	Tensile Strength, S, psi.	Brinell Number, H	Ratio, $\frac{S}{H}$
No. 1015...	Carbon	Annealed	60 000	128	470
No. 1015...	Carbon	Water-quenched and drawn	95 000	197	480
No. 1035...	Carbon	Annealed	84 000	162	520
No. 1035...	Carbon	Oil-quenched and drawn	106 000	223	470
No. 1060...	Carbon	Annealed	95 000	187	510
No. 1060...	Carbon	Oil-quenched and drawn	123 000	255	480
No. 2340...	Nickel	Oil-quenched and drawn	155 000	331	470
No. 3130...	Nickel chromium	Oil-quenched and drawn	184 000	363	510
No. 4340...	Nickel chromium molybdenum	Oil-quenched and drawn	260 000	495	520

SOME LIMITATIONS

It has been indicated that the relationship between the indentation hardness and tensile strength is very useful to the engineer. Since fatigue action accounts for a large percentage of service failures it is customary to extend the relationship between values of hardness and tensile strength to include that of fatigue strength. In this respect serious errors will arise unless all factors are taken into account.

The relation between tensile strength and hardness for the usual heat-treated steels is good, and a similar statement can be made as regards the relation between tensile strength and endurance limit.[11] It may therefore be concluded that the relation between indentation hardness and endurance limit should also be good. This, in general, is true but serious errors are apt to arise due to such interpretation. This stems from the fact that recent work has shown that the surface conditions may influence very considerably the endurance limit of the steel. Such surface conditions cannot be detected by the usual indentation hardness test as used for control purposes.

The average ratio of endurance limit to tensile strength can be taken as 0.45 to 0.5. Cazaud[12] has shown, however, that with steels which have a martensitic structure produced by quenching, this ratio may fall to as low as 0.23; while with steels having martensite produced without drastic quenching the ratio is 0.47. This reduction is due to the internal stress produced by the severe quenching operation. The indentation hardness test cannot however detect such conditions.

A further instance is that of decarburization effects. It is well known that the presence of decarburized outer layers lowers considerably the fatigue strength of steels. Batson[13] indicated a reduction of as much as 50 to 60 per cent for spring steel. This decrease in fatigue strength is due to a tensile stress developed in the extreme outer layers caused by the loss in carbon in these layers. It is possible that the presence of this decarburization could not be detected by hardness tests made on the surface. Hardness surveys[14] on taper-ground specimens would be more sensitive in this respect, but this would involve the destruction of the particular part.

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[9] S. L. Hoyt, "Metals and Alloys Data Book," Reinhold Publishing Corp., New York, N. Y. (1943).

[10] S. A. E. Handbook, Chart I, p. 358 (1943).

[11] H. J. Gough, "Fatigue of Metals," Scott, Greenwood and Son, London p. 142, (1924).

[12] R. Cazaud and L. Persoz, "La Fatigue des Metaux," Dunod, Paris p. 93, (1937).

[13] R. G. C. Batson and J. Bradley, "Fatigue Strength of Carbon and Alloy Steel Plates," *Proceedings*, Inst. of Mechanical Engrs. (London), Vol. 120, p. 301 (1931).

[14] See Discussion by H. A. Dickie, *Journal*, Iron and Steel Inst., Vol. CXXIV, No. II, p. 440 (1931).

In the case of shot blasting or peening of the surface the reverse is true, and due to a favorable compressive stress developed in the extreme outer layers the fatigue strength is considerably increased.[15] Research has shown that the thickness of such layers may be 0.006 in. This effect, in this case beneficial, cannot be detected by the usual indentation hardness test made on the surface. There is no doubt that greater attention will be given in the future towards producing favorable stresses in the outer layers in order to delay fatigue action, and the hardness test in its present form cannot help in determining the improvement so obtained.

CONCLUSIONS

It is, therefore, apparent that the indentation test as developed by Brinell has a very important place in our modern methods of material control. The papers by Williams and Hoyt which follow will describe the utility of other forms of hardness tests. The limitations which have been discussed in the present paper are of importance in so far as the correlation of indentation hardness and fatigue strength is concerned.

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[15] J. M. Lessells and W. M. Murray, "The Effect of Shot Blasting and Its Bearing on Fatigue," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 659 (1941).

APPENDIX

The following are conversion tables in frequent use to convert from hardness values obtained on one type of machine to those obtained on other types.

HARDNESS CONVERSION TABLE FOR NICKEL ALLOY STEELS.
(Approximate)

Brinell			Rockwell			
Diameter, mm., 3000-kg. Load, 10-mm. Ball	Hardness Number	Vickers or Firth	"C" Scale, 150-kg. Load, 120-deg. Cone	"B" Scale, 100-kg. Load, 1/16-in. Ball	Shore	Tensile Strength, 1000 psi.
2.20	780	1220	68	...	96	...
2.25	745	1114	67	...	94	...
2.30	712	1021	65	...	92	354
2.35	682	940	63	...	89	341
2.40	653	867	62	...	86	329
2.45	627	803	60	...	84	317
2.50	601	746	58	...	81	305
2.55	578	694	56	...	78	295
2.60	555	649	55	...	75	284
2.65	534	608	53	...	73	273
2.70	514	587	51	...	71	263
2.75	495	551	50	...	68	253
2.80	477	534	48	...	66	242
2.85	461	502	47	...	64	233
2.90	444	474	46	...	62	221
2.95	429	460	44	...	60	211
3.00	415	435	43	...	58	202
3.05	401	423	42	...	56	193
3.10	388	401	41	...	54	185
3.15	375	390	39	...	52	178
3.20	363	380	38	...	51	171
3.25	352	361	37	...	49	165
3.30	341	344	36	...	48	159
3.35	331	335	35	...	46	154
3.40	321	320	34	...	45	148
3.45	311	312	32	...	43	143
3.50	302	305	31	...	42	139
3.55	293	291	30	...	41	135
3.60	285	285	29	...	40	131
3.65	277	278	28	...	38	127
3.70	269	272	27	...	37	124
3.75	262	261	26	...	36	121
3.80	255	255	25	...	35	117
3.85	248	250	24	100	34	115
3.90	241	240	23	99	33	112
3.95	235	235	22	99	32	109
4.00	229	226	21	98	32	107
4.05	223	221	20	97	31	105
4.10	217	217	18	96	30	103
4.15	212	213	17	95	30	100
4.20	207	209	16	95	29	98
4.30	197	197	14	93	28	95
4.40	187	186	12	91	27	91
4.50	179	177	10	89	25	87
4.60	170	171	8	87	24	84
4.70	163	162	6	85	23	81
4.80	156	154	4	83	23	78
4.90	149	149	2	81	22	76
5.00	143	144	0	79	21	74
5.10	137	136	−3	77	20	71

Data compiled by Research Lab., Development and Research Div., The International Nickel Co., Inc., 67 Wall St., New York, N. Y.

Notes for Tables on pp. 809 to 811

APPROXIMATE RELATIONSHIPS BETWEEN HARDNESS VALUES DETERMINED ON "ROCKWELL" AND "ROCKWELL SUPERFICIAL" HARDNESS TESTERS AND VALUES DETERMINED ON OTHER TESTERS.*

All so-called "conversion" tables of hardness scales, including those here published, are and must be based on the assumption that the metal tested is homogeneous to a depth several times as great as the depth of the indentation, because different loads and different shapes of penetrators would, in metal not homogeneous, penetrate—or at least meet the resistance of—metal of varying hardness, depending upon the depth of the indentation, hence, no definite hardness value would actually exist and no recorded hardness value would be valid to an extent that could be confirmed by another person unless shape of penetrator and actual load applied are both specified.

These fundamentally essential specifications as to both loads and penetrators are and always have been fully covered in the recording of readings obtained on "ROCKWELL" Hardness Testers by the use of alphabetical symbols.

Conversion tables dealing with hardness can be only approximate and never mathematically exact, for it must be understood that a penetration hardness test proceeds until the specimen tested supports the applied load. Yet it is a severely cold-worked metal that actually supports the penetrator, and different metals, different alloys, and different analyses of the same type of alloy, have different cold-working properties. Nevertheless, while a conversion table cannot be mathematically exact, it is of considerable value to be able to compare different hardness scales in a general way.

Influence on Relationships, Within the B-Scale Range, by Work Hardening Behavior of Material. The indentation hardness values measured on the various scales depend on the work hardening behavior of the material during the test and this in turn depends on the degree of previous cold working of the material. The B-scale relationships in the table are based largely on annealed metals for the low values and cold-worked metals for the higher values. Therefore, annealed metals of high B-scale hardness such as austenitic stainless steels, nickel and high nickel alloys do not conform closely to these general tables. Neither do cold-worked metals of low B-scale hardness such as aluminum and the softer alloys. Special correlations are needed for more exact relationships in these cases.

* Taken from Chart 38 issued by the Wilson Mechanical Instrument Co., Inc.

** The 15-T, 30-T, 45-T, 15-N, 30-N, and 45-N values are in scales of the "ROCKWELL" Superficial Hardness Tester, a specialized form of "ROCKWELL" Tester, having lighter loads and more sensitive depth reading system, used where for one or another reason the indentation must be exceptionally shallow.

FOR HARDENED STEEL AND HARD ALLOYS.

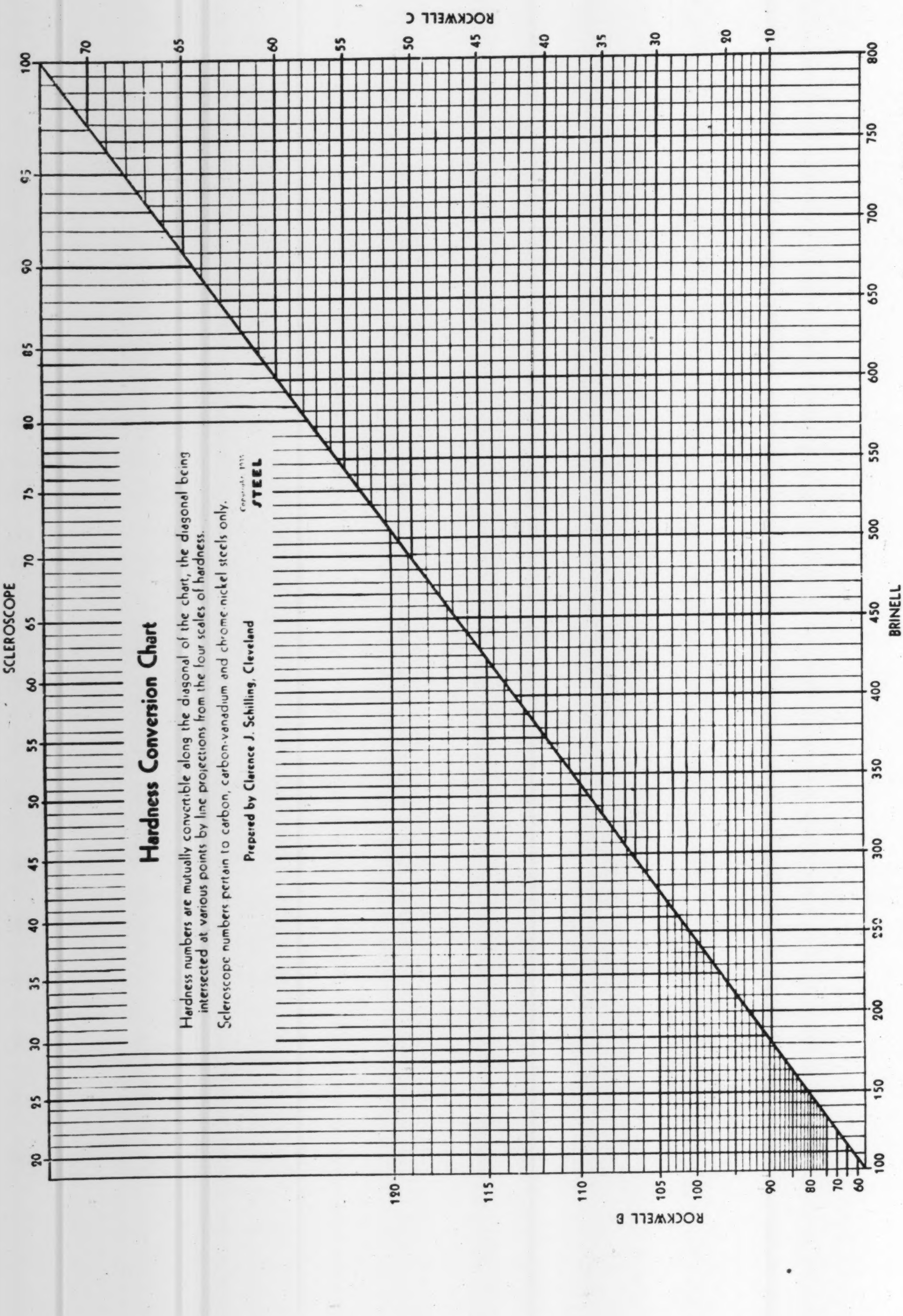
C	A	D	15-N**	30-N**	45-N**	Diamond Pyramid	Brinell	G	Tensile Strength
Rockwell Hardness Tester			Rockwell Superficial			4 sided 136-deg. Diamond Pyramid 10-kg. Load—Measurement of 2 diagonals by microscope. Hardness numbers computed by formula.	Brinell, Standard Type, 3000-kg. Load Hultgren 10-mm. Ball penetrator	Rockwell Hardness Tester, G scale, 1/16-in. Ball penetrator—150-kg. Load	Thousand psi.
C scale, "Brale" penetrator—150-kg. Load	A scale, "Braie" penetrator—60-kg. Load	D scale, "Brale" penetrator—100-kg. Load	15-N scale, "N Brale" penetrator—15-kg. Load	30-N scale, "N Brale" penetrator—30-kg. Load	45-N scale, "N Brale" penetrator—45-kg. Load				
80	92.0	86.5	96.5	92.0	87.0	1865	*Note.*—General Conversion Values on material harder than about C-70 on the ROCKWELL Hardness Tester will probably be long in dispute because as yet there are few materials hard enough to be brought into that study and because some of those few do not flow nicely under the testing load but have a tendency to crumble and others do not approach homogeneity.		INEXACT AND ONLY FOR STEEL
79	91.5	85.5	...	91.5	86.5	1787			
78	91.0	84.5	96.0	91.0	85.5	1710			
77	90.5	84.0	...	90.5	84.5	1633			
76	90.0	83.0	95.5	90.0	83.5	1556			
75	89.5	82.5	...	89.0	82.5	1478			
74	89.0	81.5	95.0	88.5	81.5	1400			
73	88.5	81.0	...	88.0	80.5	1323			
72	88.0	80.0	94.5	87.0	79.5	1245			
71	87.0	79.5	...	86.5	78.5	1160			
70	86.5	78.5	94.0	86.0	77.5	1076			
69	86.0	78.0	93.5	85.0	76.5	1004			
68	85.5	77.0	...	84.5	75.5	942			
67	85.0	76.0	93.0	83.5	74.5	894			
66	84.5	75.5	92.5	83.0	73.0	854			
65	84.0	74.5	92.0	82.0	72.0	820			
64	83.5	74.0	...	81.0	71.0	789			
63	83.0	73.0	91.5	80.0	70.0	763			
62	82.5	72.5	91.0	79.0	69.0	739	...	...	...
61	81.5	71.5	90.5	78.5	67.5	716	...	...	...
60	81.0	71.0	90.0	77.5	66.5	695	614	...	314
59	80.5	70.0	89.5	76.5	65.5	675	600	...	306
58	80.0	69.0	...	75.5	64.0	655	587	...	299
57	79.5	68.5	89.0	75.0	63.0	636	573	...	291
56	79.0	67.5	88.5	74.0	62.0	617	560	..	284
55	78.5	67.0	88.0	73.0	61.0	598	547	...	277
54	78.0	66.0	87.5	72.0	59.5	580	534	...	270
53	77.5	65.5	87.0	71.0	58.5	562	522	...	263
52	77.0	64.5	86.5	70.5	57.5	545	509	...	256
51	76.5	64.0	86.0	69.5	56.0	528	496	...	250
50	76.0	63.0	85.5	68.5	55.0	513	484	...	243
49	75.5	62.0	85.0	67.5	54.0	498	472	....	236
48	74.5	61.5	84.5	66.5	52.5	485	460	...	230
47	74.0	60.5	84.0	66.0	51.5	471	448	...	223
46	73.5	60.0	83.5	65.0	50.0	458	437	...	217
45	73.0	59.0	83.0	64.0	49.0	446	426	...	211
44	72.5	58.5	82.5	63.0	48.0	435	415	...	205
43	72.0	57.5	82.0	62.0	46.5	424	404	....	199
42	71.5	57.0	81.5	61.5	45.5	413	393	...	194
41	71.0	56.0	81.0	60.5	44.5	403	382	...	188
40	70.5	55.5	80.5	59.5	43.0	393	372	...	182
39	70.0	54.5	80.0	58.5	42.0	383	362	...	177
38	69.5	54.0	79.5	57.5	41.0	373	352	...	171
37	69.0	53.0	79.0	56.5	39.5	363	342	...	166
36	68.5	52.5	78.5	56.0	38.5	353	332	...	162
35	68.0	51.5	78.0	55.0	37.0	343	322	...	157
34	67.5	50.5	77.0	54.0	36.0	334	313	...	153
33	67.0	50.0	76.5	53.0	35.0	325	305	...	148
32	66.5	49.0	76.0	52.0	33.5	317	297	...	144
31	66.0	48.5	75.5	51.5	32.5	309	290	...	140
30	65.5	47.5	75.0	50.5	31.5	301	283	92.0	136
29	65.0	47.0	74.5	49.5	30.0	293	276	91.0	132
28	64.5	46.0	74.0	48.5	29.0	285	270	90.0	129
27	64.0	45.5	73.5	47.5	28.0	278	265	89.0	126
26	63.5	44.5	72.5	47.0	26.5	271	260	88.0	123
25	63.0	44.0	72.0	46.0	25.5	264	255	87.0	120
24	62.5	43.0	71.5	45.0	24.0	257	250	86.0	117
23	62.0	42.5	71.0	44.0	23.0	251	245	84.5	115
22	61.5	41.5	70.5	43.0	22.0	246	240	83.5	112
21	61.0	41.0	70.0	42.5	20.5	241	235	82.5	110
20	60.5	40.0	69.5	41.5	19.5	236	230	81.0	108

FOR UNHARDENED STEEL, STEEL OF SOFT TEMPER, GRAY AND

B	F	G	15-T**	30-T**	45-T**	E	H	K	A	Brinell	Brinell	Tensile Strength
Rockwell Hardness Tester			Rockwell Superficial			Rockwell Hardness Tester				Brinell, Standard Type, 500-kg. Load 10-mm. Ball penetrator	Brinell, Standard Type, 3000-kg. Load, 10-mm. Ball penetrator	Thousand psi.
B scale, 1/16-in. Ball penetrator—100-kg. Load	F scale, 1/16-in. Ball penetrator—60-kg. Load	G scale, 1/16-in. Ball penetrator—150-kg. Load	15-T scale, 1/16-in. Ball penetrator—15-kg. Load	30-T scale, 1/16-in. Ball penetrator—30-kg. Load	45-T scale, 1/16-in. Ball penetrator—45-kg. Load	E scale, ⅛-in. Ball penetrator—100-kg. Load	H scale, ⅛-in. Ball penetrator—60-kg. Load	K scale, ⅛-in. Ball penetrator—150-kg. Load	A scale, "Brale" penetrator—60-kg. Load			
100	...	82.5	93.0	82.0	72.0	...	...	...	61.5	201	240	116
99	...	81.0	92.5	81.5	71.0	...	...	...	61.0	195	234	112
98	...	79.0	...	81.0	70.0	...	...	...	60.0	189	228	109
97	...	77.5	92.0	80.5	69.0	...	...	...	59.5	184	222	106
96	...	76.0	...	80.0	68.0	...	...	...	59.0	179	216	103
95	...	74.0	91.5	79.0	67.0	...	...	...	58.0	175	210	101
94	...	72.5	...	78.5	66.0	...	...	...	57.5	171	205	98
93	...	71.0	91.0	78.0	65.5	...	...	...	57.0	167	200	96
92	...	69.0	90.5	77.5	64.5	...	...	100	56.5	163	195	93
91	...	67.5	...	77.0	73.5	...	...	99.5	56.0	160	190	91
90	...	66.0	90.0	76.0	62.5	...	...	98.5	55.5	157	185	89
89	...	64.0	89.5	75.5	61.5	...	...	98.0	55.0	154	180	87
88	...	62.5	...	75.0	60.5	...	...	97.0	54.0	151	176	85
87	...	61.0	89.0	74.5	59.5	...	...	96.5	53.5	148	172	83
86	...	59.0	88.5	74.0	58.5	...	...	95.5	53.0	145	169	81
85	...	57.5	...	73.5	58.0	...	...	94.5	52.5	142	165	80
84	...	56.0	88.0	73.0	57.0	...	...	94.0	52.0	140	162	78
83	...	54.0	87.5	72.0	56.0	...	...	93.0	51.0	137	159	77
82	...	52.5	...	71.5	55.0	...	...	92.0	50.5	135	156	75
81	...	51.0	87.0	71.0	54.0	...	...	91.0	50.0	133	153	74
80	...	49.0	86.5	70.0	53.0	...	...	90.5	49.5	130	150	72
79	...	47.5	...	69.5	52.0	...	...	89.5	49.0	128	147	Even for steel, Tensile Strength relation to hardness is inexact, unless determined for specific material.
78	...	46.0	86.0	69.0	51.0	...	...	88.5	48.5	126	144	
77	...	44.0	85.5	68.0	50.0	...	...	88.0	48.0	124	141	
76	...	42.5	...	67.5	49.0	...	...	87.0	47.0	122	139	
75	99.5	41.0	85.0	67.0	48.5	...	...	86.0	46.5	120	137	
74	99.0	39.0	...	66.0	47.5	...	...	85.0	46.0	118	135	
73	98.5	37.5	84.5	65.5	46.5	...	...	84.5	45.5	116	132	
72	98.0	36.0	84.0	65.0	45.5	...	...	83.5	45.0	114	130	
71	97.5	34.5	...	64.0	44.5	100	...	82.5	44.5	112	127	
70	97.0	32.5	83.5	63.5	43.5	99.5	...	81.5	44.0	110	125	
69	96.0	31.0	83.0	62.5	42.5	99.0	...	81.0	43.5	109	123	
68	95.5	29.5	...	62.0	41.5	98.0	...	80.0	43.0	107	121	
67	95.0	28.0	82.5	61.5	40.5	97.5	...	79.0	42.5	106	119	
66	94.5	26.5	82.0	60.5	39.5	97.0	...	78.0	42.0	104	117	
65	94.0	25.0	...	60.0	38.5	96.0	...	77.5	...	102	116	
64	93.5	23.5	81.5	59.5	37.5	95.5	...	76.5	41.5	101	114	
63	93.0	22.0	81.0	58.5	36.5	95.0	...	75.5	41.0	99	112	
62	92.0	20.5	...	58.0	35.5	94.5	...	74.5	40.5	98	110	
61	91.5	19.0	80.5	57.0	34.5	93.5	...	74.0	40.0	96	108	
60	91.0	17.5	...	56.5	33.5	93.0	...	73.0	39.5	95	107	
59	90.5	16.0	80.0	56.0	32.0	92.5	...	72.0	39.0	94	106	
58	90.0	14.5	79.5	55.0	31.0	92.0	...	71.0	38.5	92	104	
57	89.5	13.0	...	54.5	30.0	91.0	...	70.5	38.0	91	103	
56	89.0	11.5	79.0	54.0	29.0	90.5	...	69.5	...	90	101	
55	88.0	10.0	78.5	53.0	28.0	90.0	...	68.5	37.5	89	100	
54	87.5	8.5	...	52.5	27.0	89.5	...	68.0	37.0	87	...	
53	87.0	7.0	78.0	51.5	26.0	89.0	...	67.0	36.5	86	...	
52	86.5	5.5	77.5	51.0	25.0	88.0	...	66.0	36.0	85	...	
51	86.0	4.0	...	50.5	24.0	87.5	...	65.0	35.5	84	...	
50	85.5	2.5	77.0	49.5	23.0	87.0	...	64.5	35.0	83	...	

MALLEABLE CAST IRON AND MOST NON-FERROUS METAL.

B	F	G	15-T**	30-T**	45-T**	E	H	K	A	Brinell
Rockwell Hardness Tester			Rockwell Superficial			Rockwell Hardness Tester				
B scale, 1/16-in. Ball penetrator—100-kg. Load	F scale, 1/16-in. Ball penetrator—60-kg. Load	G scale, 1/16-in. Ball penetrator—150-kg. Load	15-T scale, 1/16-in. Ball penetrator—15-kg. Load	30-T scale, 1/16-in. Ball penetrator—30-kg. Load	45-T scale, 1/16-in. Ball penetrator—45-kg. Load	E scale, ⅛-in. Ball penetrator—100-kg. Load	H scale, ⅛-in. Ball penetrator—60-kg. Load	K scale, ⅛-in. Ball penetrator—150-kg. Load	A scale, "Brale" penetrator—60-kg. Load	Brinell, Standard Type, 500-kg. Load, 10-mm. Ball penetrator
50	85.5	2.5	77.0	49.5	23.0	87.0	...	64.5	35.0	83
49	85.0	1.0	76.5	49.0	22.0	86.5	...	63.5	...	82
48	84.5	...	...	48.5	20.5	85.5	...	62.5	34.5	81
47	84.0	...	76.0	47.5	19.5	85.0	...	61.5	34.0	80
46	83.0	...	75.5	47.0	18.5	84.5	...	61.0	33.5	...
45	82.5	...	...	46.0	17.5	84.0	...	60.0	33.0	79
44	82.0	...	75.0	45.5	16.5	83.5	...	59.0	32.5	78
43	81.5	...	74.5	45.0	15.5	82.5	...	58.0	32.0	77
42	81.0	...	...	44.0	14.5	82.0	...	57.5	31.5	76
41	80.5	...	74.0	43.5	13.5	81.5	...	56.5	31.0	75
40	79.5	...	73.5	43.0	12.5	81.0	...	55.5	...	...
39	79.0	...	...	42.0	11.0	80.0	...	54.5	30.5	74
38	78.5	...	73.0	41.5	10.0	79.5	...	54.0	30.0	73
37	78.0	...	72.5	40.5	9.0	79.0	...	53.0	29.5	72
36	77.5	...	...	40.0	8.0	78.5	100	52.0	29.0	...
35	77.0	...	72.0	39.5	7.0	78.0	99.5	51.5	28.5	71
34	76.5	...	71.5	38.5	6.0	77.0	99.0	50.5	28.0	70
33	75.5	...	...	38.0	5.0	76.5	...	49.5	...	69
32	75.0	...	71.0	37.5	4.0	76.0	98.5	48.5	27.5	...
31	74.5	...	...	36.5	3.0	75.5	98.0	48.0	27.0	68
30	74.0	...	70.5	36.0	2.0	75.0	...	47.0	26.5	67
29	73.5	...	70.0	35.5	1.0	74.0	97.5	46.0	26.0	...
28	73.0	...	...	34.5	...	73.5	97.0	45.0	25.5	66
27	72.5	...	69.5	34.0	...	73.0	96.5	44.5	25.0	...
26	72.0	...	69.0	33.0	...	72.5	...	43.5	24.5	65
25	71.0	...	...	32.5	...	72.0	96.0	42.5	...	64
24	70.5	...	68.5	32.0	...	71.0	95.5	41.5	24.0	...
23	70.0	...	68.0	31.0	...	70.5	...	41.0	23.5	63
22	69.5	...	...	30.5	...	70.0	95.0	40.0	23.0	...
21	69.0	...	67.5	29.5	...	69.5	94.5	39.0	22.5	62
20	68.5	...	...	29.0	...	68.5	...	38.0	22.0	...
19	68.0	...	67.0	28.5	...	68.0	94.0	37.5	21.5	61
18	67.0	...	66.5	27.5	...	67.5	93.5	36.5	...	...
17	66.5	...	...	27.0	...	67.0	93.0	35.5	21.0	60
16	66.0	...	66.0	26.0	...	66.5	...	35.0	20.5	...
15	65.5	...	65.5	25.5	...	65.5	92.5	34.0	20.0	59
14	65.0	...	...	25.0	...	65.0	92.0	33.0	...	...
13	64.5	...	65.0	24.0	...	64.5	...	32.0	...	58
12	64.0	...	64.5	23.5	...	64.0	91.5	31.5	...	...
11	63.5	...	...	23.0	...	63.5	91.0	30.5	...	...
10	63.0	...	64.0	22.0	...	62.5	90.5	29.5	...	57
9	62.0	...	...	21.5	...	62.0	...	29.0	...	...
8	61.5	...	63.5	20.5	...	61.5	90.0	28.0	...	...
7	61.0	...	63.0	20.0	...	61.0	89.5	27.0	...	56
6	60.5	...	...	19.5	...	60.5	...	26.0	...	...
5	60.0	...	62.5	18.5	...	60.0	89.0	25.5	...	55
4	59.5	...	62.0	18.0	...	59.0	88.5	24.5	...	...
3	59.0	...	...	17.0	...	58.5	88.0	23.5	...	...
2	58.0	...	61.5	16.5	...	58.0	...	23.0	...	54
1	57.5	...	61.0	16.0	...	57.5	87.5	22.0	...	...
0	57.0	...	...	15.0	...	57.0	87.0	21.0	...	53



Hardness Conversion Chart

Hardness numbers are mutually convertible along the diagonal of the chart, the diagonal being intersected at various points by line projections from the four scales of hardness.

Scleroscope numbers pertain to carbon, carbon-vanadium and chrome-nickel steels only.

Prepared by Clarence J. Schilling, Cleveland

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STEEL

Supplement to STEEL, Sept. 23, 1935

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NOTE.—Very complete bibliographies are contained in "Hardness of Metals" by H. O'Neill and in "Hardness and Hardness Measurements" by S. R. Williams.

[See General Discussion, page 843.—ED.]

PRESENT TYPES OF HARDNESS TESTS

By S. R. Williams[1]

In the upward progress of man some measure of the hardness of a body became necessary. The mineralogist wanted to classify his minerals, the metallurgist needed some means whereby he could catalog his metals as to their relative powers of penetration. This growth in the development of instruments for measuring hardness makes an interesting story in the annals of discovery and invention.

For convenience in classifying the more widely used hardness testers, the following arbitrary divisions have been made: (1) scratch, (2) indentation, (3) rebound, (4) portable, and (5) magnetic.

In the presentation of this paper at the Annual Meeting of the Society each group of hardness testers was discussed at considerable length and with some attention to the steps in developing the modern instruments. Due to a desire to have the published paper as concise as possible, only what appears to the author to be the outstanding and modern instruments are discussed.

SCRATCH METHOD

Bierbaum's Standard Instrument or the Microcharacter

From the standpoint of reproducibility and its adaptability for research, Bierbaum's instrument stands at the top.

Figure 1 shows the tester arranged for actual use.

The most important part of the instrument is the small diamond, ground with precision to form a cutting point, which has the form of the corner of a cube. Figure 2 shows how the rough diamond is attached to a stylus while it is being ground down to the desired form. Bierbaum has found that the diamond point, shaped like the corner of a cube, is so reproducible that an instrument designed by a foreign firm gave faithful and consistent repetition of readings made with an instrument built here in the United States. In this instrument there is a constant load, usually 3 g., applied to the diamond; and the width of the cut, as measured by the micrometer microscope shown in Fig. 1, is taken as a measure of the hardness of the substance tested. This is expressed by the formula, $K = 10^4/\lambda^2$, where K is the microhardness number and λ is the width of the cut made by the diamond measured in microns. The number 10^4 is an arbitrary one used to avoid fractional microhardness numbers. These values, all worked out, are given in tables accompanying the instrument. After a well-shaped corner has been formed, it is attached to a jewel spring as shown in Fig. 3. The complete jewel spring is illustrated in Fig. 4. Figure 5 shows an enlarged view of one of these diamonds after grinding and before it was used. This gives an opportunity to follow the wear of the cutting corner and to see whether it is in any way rounded off or broken by use.

The small crank K in Fig. 1 operates a screw whereby the surface of the

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[1] Fayerweather Laboratory of Physics, Amherst College, Amherst, Mass.

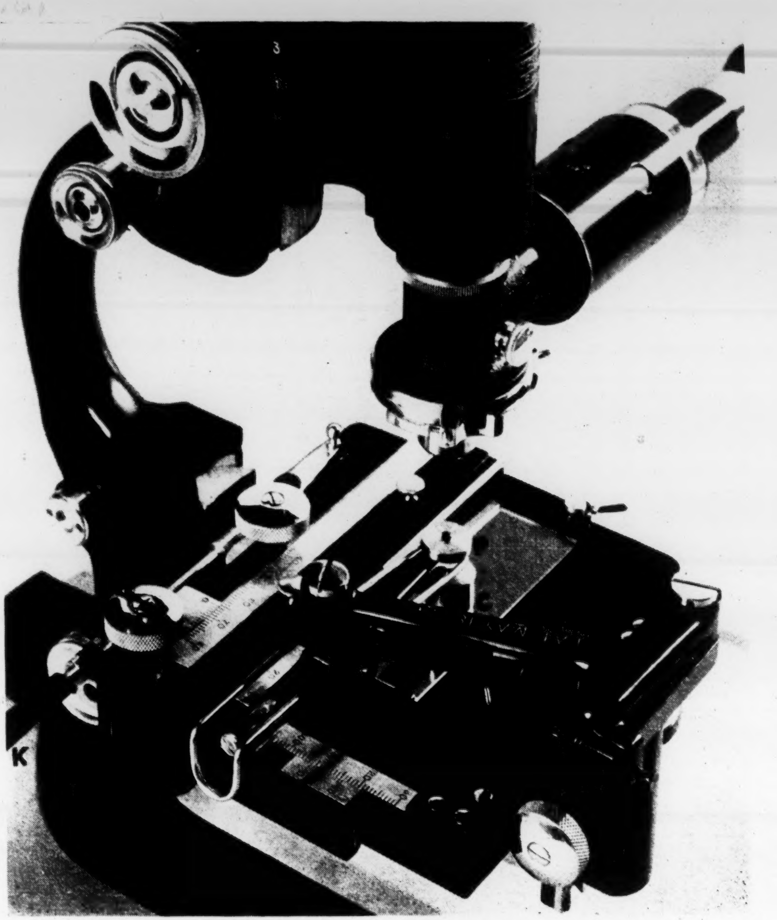


FIG. 1.—Bierbaum's Standard Instrument Attached to a Microscope with Which the Width of Scratch is Measured.

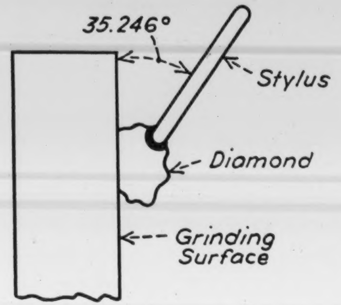

35.246°
Stylus
Diamond
Grinding Surface

FIG. 2.—Position of Stylus with Respect to Grinding Surface in Order to Form the Proper Cutting Edge on the Diamond.

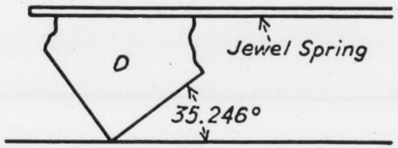

D
Jewel Spring
35.246°

FIG. 3.—Showing How the Cut Diamond is Mounted on Its Spring.

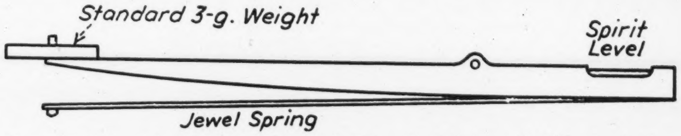

Standard 3-g. Weight
Spirit Level
Jewel Spring

FIG. 4.—Mounting of the Diamond Edge in Bierbaum's Standard Instrument in Order to Secure Uniform Pressure on the Cutting Edge.

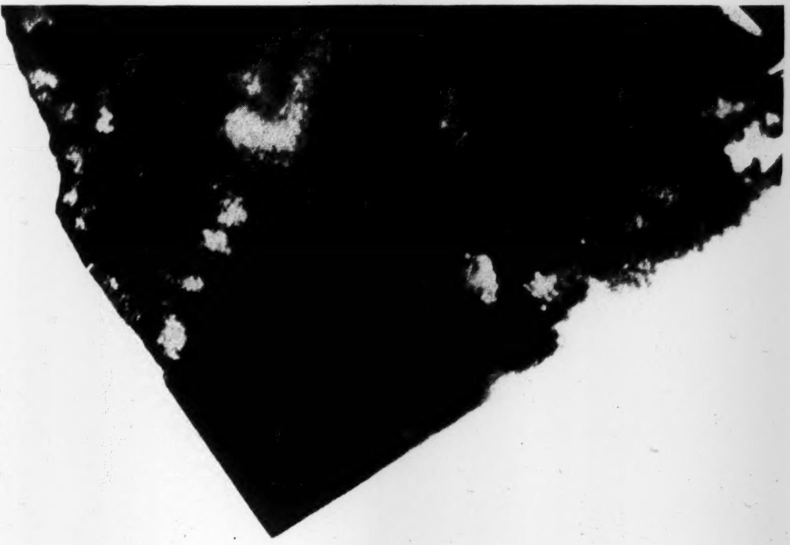


FIG. 5.—Profile of the Cutting Edge of the Diamond as Used in Bierbaum's Standard Instrument.

specimen *C* being tested is moved steadily and slowly under the diamond point *P*. As the surface moves along, the diamond point makes a cut in the surface like that shown in Fig. 6 where the cut leads across a piece of bronze in which is imbedded a crystal of tin oxide. The difference in width of cut between the bronze and the tin oxide is apparent.

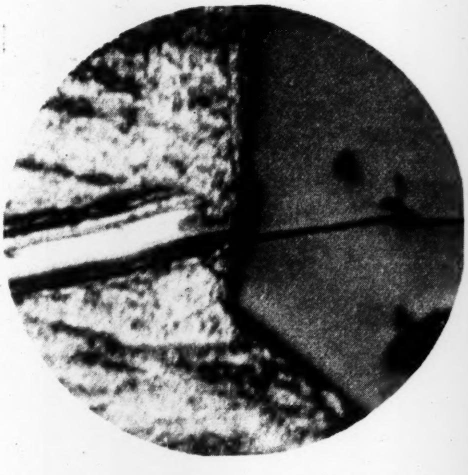


FIG. 6.—Microcut Made by the Bierbaum Standard Instrument Across a Piece of Bronze in Which Is Imbedded a Crystal of Tin Oxide.

It should be emphasized that the Bierbaum standard instrument when properly used makes a cut instead of a scratch. That difference may be brought out by studying Figs. 7, 8, and 9. Figure 7 shows a cut in a glass surface when the regular load of 3 g. was carried on the diamond point. When 9 g. were used as a load there resulted a shearing and chipping as in Fig. 8 which is the beginning of a real scratch as made by a diamond glass cutter shown in Fig. 9.

The Bierbaum instrument is preeminently a research tool. I take this opportunity to express my appreciation of Mr. Bierbaum's cooperation in furnishing a nonmagnetic jewel spring for the instrument which I used in measuring the change in hardness when a piece of steel is worked magnetically. As shown in Fig. 10 the width of cut in the magnetically worked material is for a softer material than in the aged con-

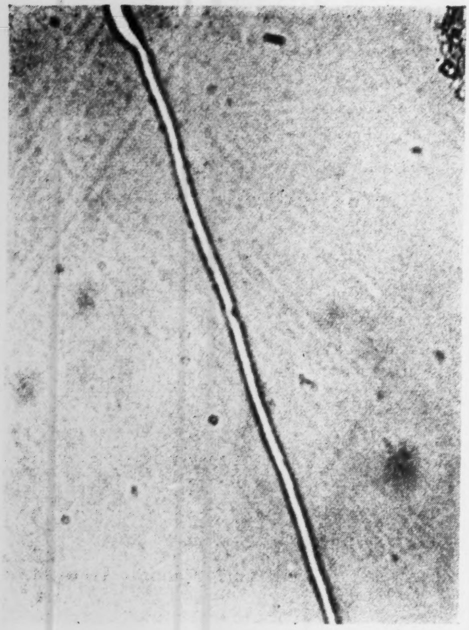


FIG. 7.—Microcut Made by the Bierbaum Standard Instrument on a Surface of Glass with a Load of 3 g.

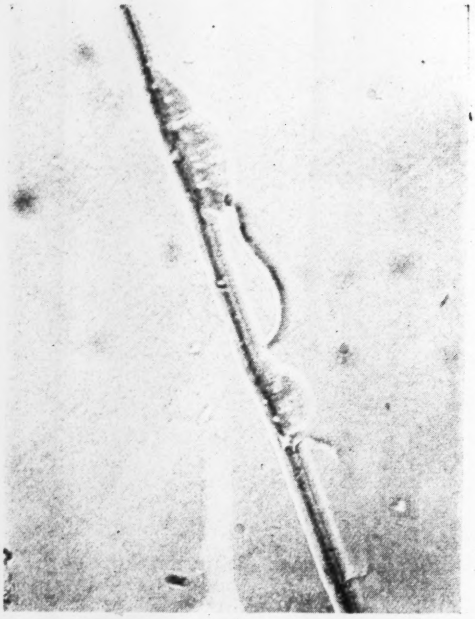


FIG. 8.—Microcut Made by the Bierbaum Standard Instrument on a Surface of Glass with a Load of 9 g. This load is too heavy.

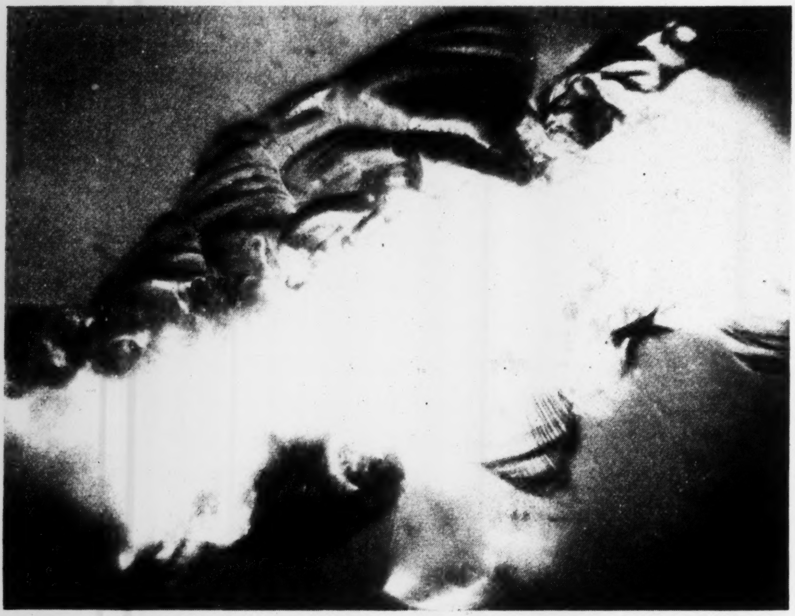


FIG. 9.—Compared with Fig. 7 This Shows the Difference Between a Cut and a Scratch.

dition. In the process of aging, the atoms in the surface of the steel sample have settled into a firmer surface layer than they are when they are shaken up by repeated reversals of a magnetic field which is slowly decreasing. Such a process of shaking up is what we do when demagnetizing a piece of steel. That surface layers of magnetic materials do play an important part in their behavior was shown by the author in some work[2] on piles of nickel plates.

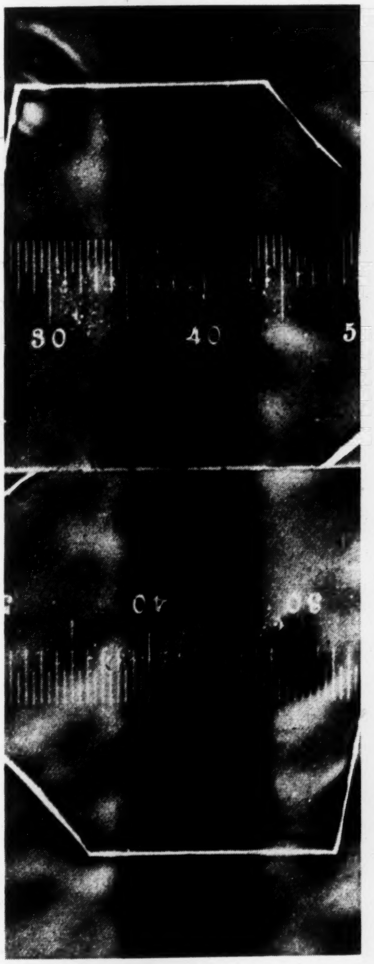
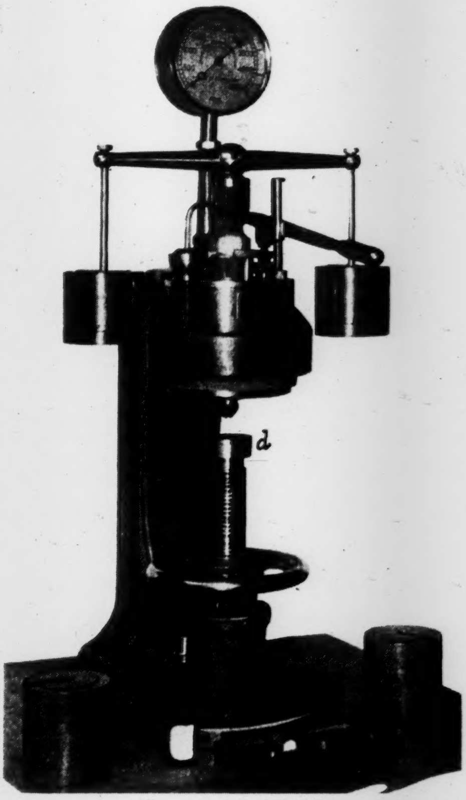


FIG. 10.—A Microcut Made on Highly Polished Steel.

The upper half of the microcut is on magnetically worked steel. The cut is wider than the one in the lower half which was on the same piece of steel before being magnetically worked. The two cuts were made side by side.

Precautions.—In using the Bierbaum standard or microcut instrument, care must be taken that the point making the cut does not receive blows. A diamond point is easily shattered. Once the point is injured no further comparisons can be made with the readings which were made before the injury. One must also remember that the sharp corner of the diamond used as a cutting point can



(Courtesy of Steel City Testing Laboratory)

FIG. 11.—One Interpretation of the Brinell Hardness Tester. Style "A" hand-operated Brinell machine.

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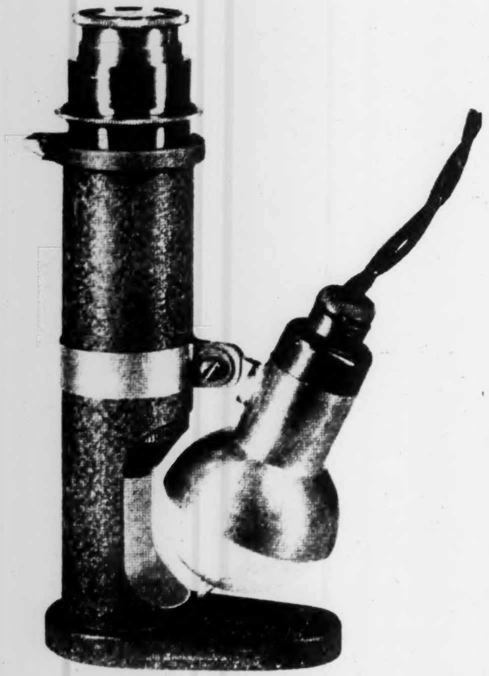
[2] S. R. Williams, "The Effect of Transverse Joints on the Magnetic Induction in Nickel," *Journal*, Optical Soc. of America, Vol. 10, pp. 109-118 (1925).

have an enormous pressure exerted on its point by a comparatively small load.

Again, care must be taken that the diamond point does not chatter when the cut is made on the substance tested.



(Courtesy of Pittsburgh Instrument and Machine Co.)

FIG. 12.—Microscope for Measuring the Diameter of the Indentation Made by the Brinell Hardness Tester.

PENETRATION OR INDENTATION METHODS

Brinell Hardness Tester:

J. A. Brinell, a Swedish engineer, gave a great impetus to the development of the technique of modern hardness testing. In describing the instruments to be reviewed in this section, there is such a similarity in the general mechanism whereby some form of penetrator can have a load impressed on it and cause the penetrator to make a permanent deformation in the surface of the test specimen, that a single description for all of them will suffice.

In the Brinell instrument, Fig. 11, the test specimen is brought up under the indenter by means of the press table *d*. At a proper height of the specimen an hydraulic press forces a plunger downward which carries a steel ball at its lower end. As the steel ball contacts the surface of the specimen it is forced into the material until a definite pressure is attained in the hydraulic press. The steel ball makes an impression in the surface of the specimen, the diameter of which is used as a measure of the hardness of the material. By means of a formula,

$$H = \frac{P}{\frac{\pi D}{2}\left(D - \sqrt{D^2 - d^2}\right)}$$

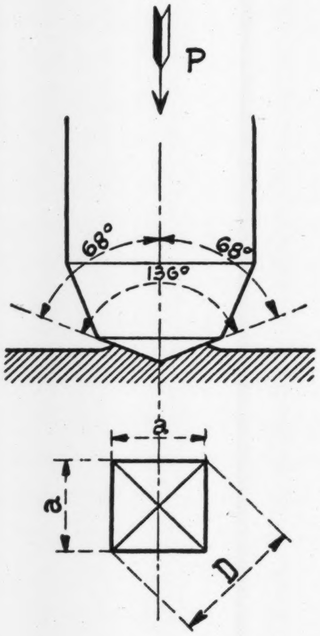


FIG. 13.—Showing how the Angle at the Apex of the Diamond Pyramid Indenter Was Determined for Use in the Vickers Pyramid Hardness Tester. *D* is the length measured.

the relation between the hardness H and the diameter of the impression d has been worked out in a chart for a given size steel ball whose diameter is D. As soon as the impression has been made in the specimen, its diameter is measured by means of an illuminated Brinell microscope and from the chart the hardness number is found directly. This microscope is shown in Fig. 12. The Brinell instrument is an old, trustworthy device for measuring hardness and is still widely used in commercial plants. For measuring very hard substances there are specially hardened steel balls such as (1) Hultgren's extra hard steel balls, (2) special tungsten-carbide balls, and (3) hemispherical diamond indenters.

Instead of the spherical indenter ordinarily used on the Brinell machine, a diamond pyramid indenter with a 136-deg. angle between opposite faces may be used. The machine may then serve as a Vickers pyramid hardness tester. Instead of measuring the diameter of the spherical indentation, the diagonal D of the square indentation is measured as shown in Fig. 13. Ludwik's cone indenter could also be used. His indenter was a cone with a solid angle of 90 deg. With this angle, the depth and diameter were the same and the hardness number was given by the equation:

$$\text{Hardness} = \frac{\text{Load}}{\text{Area of imprint}}$$

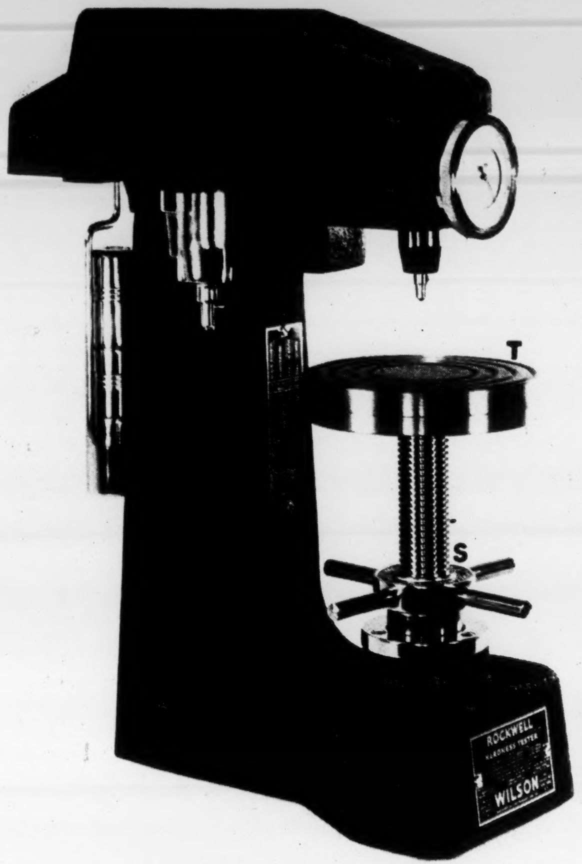
which due to the geometrical similarity of the imprints, the hardness numbers obtained are independent of the magnitude of the load used and of the size of the imprint.

It is not difficult to substitute any form of indenter on the various indentation hardness testers. The author has compared various indenters on the same Rockwell machine.

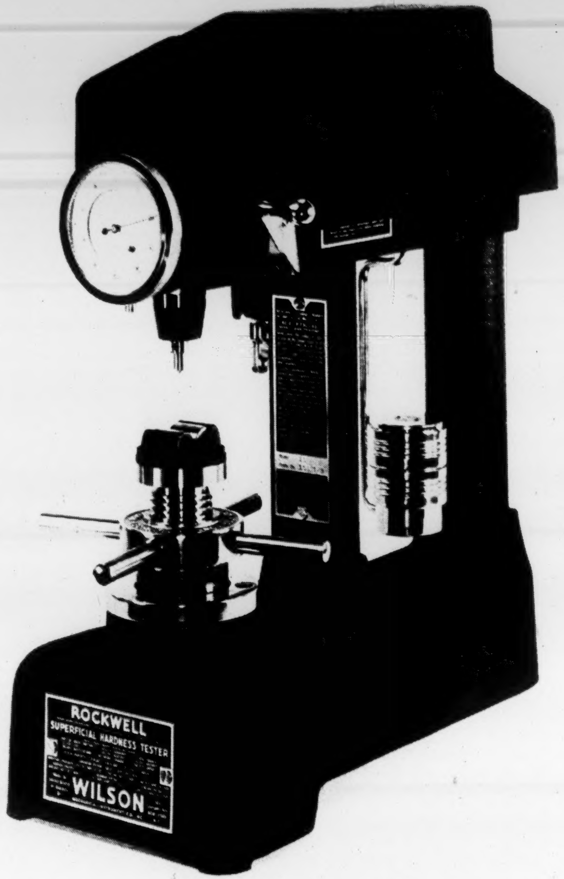
Rockwell's Instrument:

The Rockwell hardness measuring device has attained an enviable reputation because of its speed, accuracy and wide range of adaptability. (See Fig. 14.) Brinell based his tests on the diameter of the indentation made by a steel ball or a diamond hemisphere. Rockwell based his tests on the depth of the indentation of the spherical penetrator. This latter procedure increases the speed because a dial indicator can be attached for measuring the depth directly and does not require the second measuring process which the Brinell instrument does in requiring the diameter of the indentation.

If the relation between depth of indentation and hardness holds, that is, if the law of similarity holds, then a wide range of hardness testing may be set up by varying both the size of the ball and the major load employed. The Rockwell instrument has 15 or more ranges of scales for hardness testing. It is always essential that in giving Rockwell hardness numbers that the scale symbol should always be added. In order to have the penetrator firmly seated on the surface, a minor load is applied to the indenter and the scale set to zero. Next a major load is applied, and the increment in depth due to increment in load is the linear measurement that forms the basis of Rockwell hardness number readings. An outstanding feature of the Rockwell instrument is its full floating frictionless plunger rod system, to the bottom of which the steel ball or diamond indenter is attached. This is a particularly important improvement for the Rockwell superficial hardness tester shown in Fig. 15. The force necessary to overcome the friction of the plunger plays a larger rôle for the superficial hardness tester than in the regular machines. In passing, it might be stated that the instrument


T
S
ROCKWELL
HARDNESS TESTER
WILSON

(Courtesy of Wilson Mechanical Instrument Co., Inc.)
FIG. 14.—Rockwell Hardness Tester.


ROCKWELL
SUPERFICIAL HARDNESS TESTER
WILSON

(Courtesy of Wilson Mechanical Instrument Co., Inc.)
FIG. 15.—Rockwell's Superficial Hardness Tester.

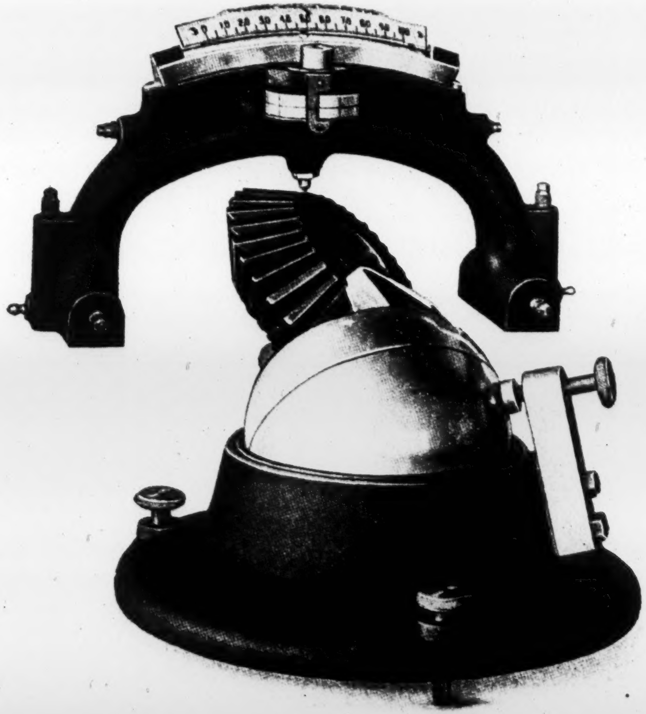


FIG. 16.—Herbert's Pendulum Hardness Tester on a Special Operating Stand for Holding Irregular Shaped Objects.

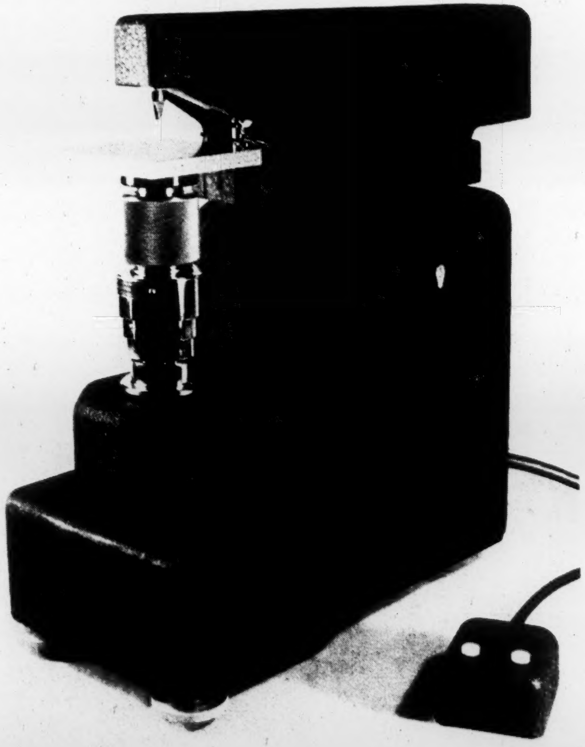


FIG. 17.—The Tukon Tester. Knoop's Hardness Tester. A tester offering some interesting possibilities in research.

shown in Fig. 15 is based on the same principle as the standard machine, that is, the increment in depth which goes with increment in load. The machine finds a wide use in measuring the hardness of thin, hard steel such as one finds in razor blades, case hardening, and rolled sheet metal.

Precautions for Indentation Hardness Testers:

On samples of soft steel or other soft metals the test specimens must not be too thin; otherwise the deformations come up against the press table. Neither must the specimens be so small as to allow a flow of metal at the edges when the ball or other form of indenter sinks in. One has more confidence in his readings if the surface of the metal tested is polished. For those indenters using depth as a measure of hardness, there must be no sinking of the specimen on the press table.

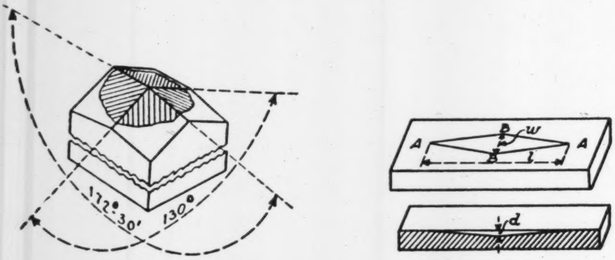


FIG. 18.—The Form of the Knoop Diamond Indenter Is Shown in This Illustration.

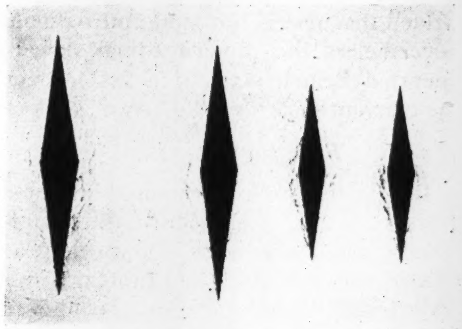
Herbert's Pendulum Hardness Tester:

This consists of an inverted U-shaped casting weighing 2 kg. (See Fig. 16.) In the testing position it rests on a 1-mm. steel ball. When placed on glass and set to rocking back and forth it can be adjusted to rock 10 complete oscillations in 100 sec., and 100 is therefore chosen as the maximum or standard value. Inasmuch as the center of gravity of this 2-kg. mass lies 0.1 mm. below the center of the steel ball on which it rocks it becomes in a way a compound pendulum and when set to rocking on a softer material it rocks more rapidly than on a hard substance like glass, because the restoring torque is greater. The length of time for 10 complete vibrations is therefore taken as a measure of the hardness of the material on which the pendulum rocks. As the pendulum rocks it really rolls on the surface of the steel ball. If there is any tendency for the ball's surface to slip on the test specimen, it is customary to use a steel ball etched in acid.

This is distinctly a research tool and needs to have an expert handle it. In fact Herbert himself seemed to be the one who got the best result out of it. The instrument must be free of vibration and set to oscillate in a space protected from air currents. It should have a case built about it like the one around a fine analytical balance. Naturally, the imprint of the steel ball is an elongated indentation.

Knoop's Instrument:

Figure 17 shows this newest of hardness testers in this class. There are some excellent features in the lever system whereby the load is applied to the indenter, but the most unique feature of all is the form of the diamond indenter itself, shown in Fig. 18. The length of the longest diagonal is chosen as the



(*Courtesy of National Bureau of Standards*)

FIG. 19.—Form of Indentations Made by Knoop Indenter on Stainless Steel. Loads of 4 and 2 kg., respectively.

basis by which the hardness number is obtained. When an indentation is made as in Fig. 19. it will be noted that the greatest strain comes along the short diagonal and least along the long diagonal. The greatest part of the recovery of the projected area of the indentation lies along the line *B-B* in Fig. 18. The hardness number is expressed by the equation

Indentation reading (hardness)

$$= \frac{\text{Load}}{\text{Area of unrecovered projected area}}$$

$$I = \frac{L}{A_p} = \frac{L}{l^2 C_p}$$

where:

L = load in kilograms,

A_p = unrecovered projected area of the indentation in square millimeters,

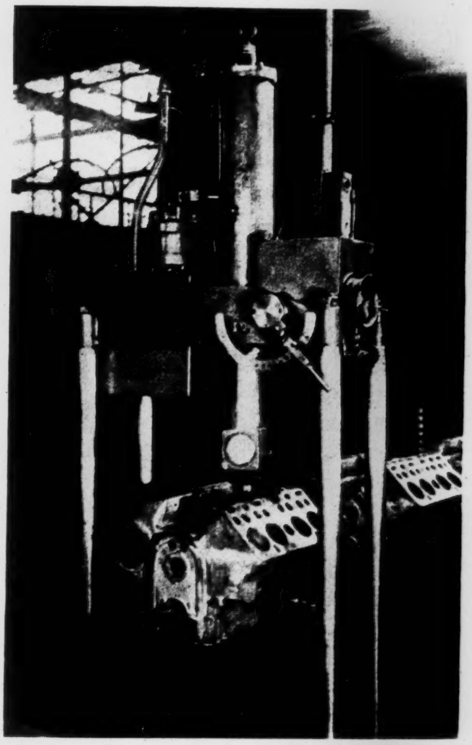
l = measured length of the long diagonal of the indentation in millimeters, and

C_p = constant relating l to the projected area.

With this form of indenter it has been possible to make a measurable indentation on diamond itself.

Precautions:

As a result of the war there has been an urge to step up production in the manufacture of war materials. This goes back to inspection of materials as well. As a result many devices have been called into existence for acceptance inspection which are really time-saving gadgets. This is particularly true of power-operated Brinell machines. To those who are familiar with the regular Brinell machine it is interesting to see how these machines are now being used on the inspection line. Figure 20 shows a line of engine blocks coming along under the indenter of one of these power-operated Brinell machines. The direct reading feature is used and no microscope



(*Courtesy of Detroit Testing Machine Co.*)

FIG. 20.—Powered Brinell Machines. Conveyor type used on cylinder block.

is required, except for an occasional check on the tolerance setting.

One firm described their setup in the following way:

"The load control valves are so arranged that the ball contacts the work, and approximately half the test load (1500 kg.) is used as a preload. At this point, the indicating gage on the front of the machine becomes operative and further penetration, beyond the preload point, is registered. The purpose of the preload is to break down surface conditions and 'set' the specimen firmly on the anvil or support.

"Due to decarburization, irregularities, contour, etc., of the work encountered, it is not advisable to place the actual hardness numbers on the dial. The actual hardness numbers would be correct only on ground, flat surfaces. Actual numbers would be misleading. As a result a dial graduated from 0 to 100 with no definite values is used.

"In setting the tolerance hands, a spot is ground or polished on the first piece to be tested. It is then placed in the machine and a test made and read in the conventional manner. This accurately determines the actual hardness. The part is then put back into the machine and a second test made on the rough surface immediately adjacent to the first and the dial reading noted. You now have a dial indication corresponding to the hardness with surface conditions automatically taken into consideration. It is then a simple matter to set the tolerance hands and the desired limits."

There has been considerable misunderstanding in regard to the so-called direct-reading machines. In the strict sense of the word, it is direct reading only on a comparison basis, but it works well as a production check. Of course the standard 3000-kg. load is applied and the microscope may be used at any time. While the operation of the conventional Brinell machine is not long and tedious, nevertheless the direct-reading, power-operated Brinell machine is faster than the conventional form.

REBOUND METHOD

It is in the group of rebound hardness testers that we need a great deal of research. It is also in this group that, as it seems to the author, our most progressive ideas have originated. I like to think of the Frenchman Martel as the pioneer in this group. Over forty years ago Martel showed that the volume of the indentation produced by the falling hammer is proportional to the height of fall and the mass of the hammer and independent of the shape. Lea in discussing Martel's work derives the following expression for the impact hardness number:

$$H_E = \frac{\text{Kinetic energy}}{\text{Volume of indentation}}$$

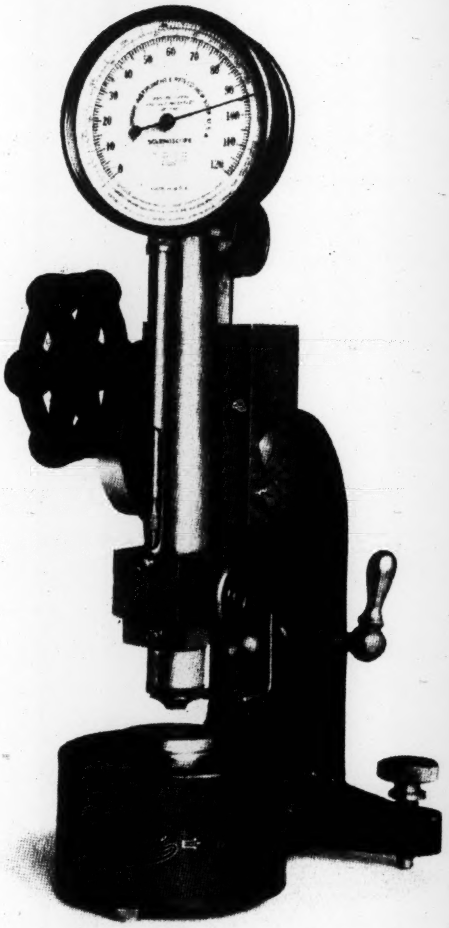
$$= \frac{\text{Force} \times \text{Length}}{\text{Area} \times \text{Length}} = \text{Stress}$$

The hardness number thus obtained is of the dimensions of a stress.

Shore's Standard Dial Recording Scleroscope:

While not discussed under portable instruments, the Shore instrument does enjoy a portability not possessed by other instruments. The instrument is shown in Fig. 21. Within the upright tube carrying the dial is a tup or diamond-tipped hammer shown in Fig. 22; and in Fig. 23 will be found a profile of the diamond. When the instrument is set for a reading the hammer is raised a certain distance and allowed to fall. The height of rebound is then made the basis for a scale of hardness numbers which are read from the face of the dial.

Actually, "the scale, against which the hammer rebounds are measured, consists



(*Courtesy of Shore Instrument and Manufacturing Co.*)

FIG. 21.—The Standard Shore Scleroscope.

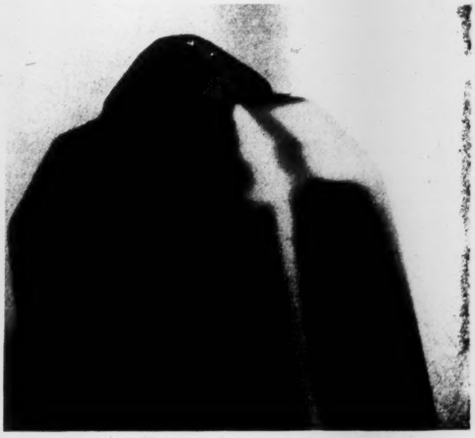


FIG. 23.—The Diamond Tip on the Hammer of a Standard Shore Scleroscope.

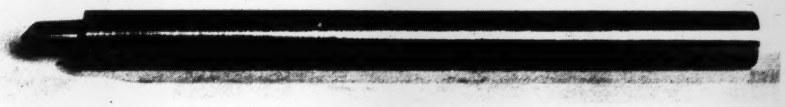


FIG. 22.—The Hammer from a Standard Shore Scleroscope.

of units which are determined by dividing the average rebound from quenched pure high-carbon steels into 100 equal parts. These rebounds run from 95 to 105 divisions." The scale is continued higher than 100 to include metals having exceptionally high or super hardness.

Precautions.—The instrument must be levelled as accurately as possible so that the hammer may fall with minimum friction. The instrument should be free from lateral movements and shocks. Do not let hammer fall on anvil of instrument. Keep the anvil in good shape. Note precaution to be found on page 446 of "Hardness and Hardness Measurements" by the author.[3]

This instrument has been very popular where a rapid check on production is needed. Comparison between hardness as found with the Shore instrument and with a penetration method shows that the two methods are not measuring the same thing. What do they measure?

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[3] S. R. Williams, "Hardness and Hardness Measurements," Am. Soc. Metals, p. 446 (1942).

Roudié's Impact Method:

About 1930, Roudié[4] came out with an interesting study of the dynamical method of hardness testing and believes he can reduce hardness measurement to the fundamental units of length, mass, and time. Landau[5] in a very recent book has reviewed Roudié's work at some length and because of space in this paper the reader is referred to this book. Roudie's tester is quite similar to the Shore instrument.

PORTABLE INSTRUMENTS

The Autopunch:

The autopunch is a convenient type of portable machine. One of the best has been put out by Rudge-Whitworth Inc. of Coventry, England. (See Fig. 24.) This instrument finds a release for the internal spring by means of a blow from a hammer applied to the projecting boss at the top of the device. The diameter of the impression is then measured as in the Brinell method.

The Barber-Colman Quick-Check Portable Hardness Tester (Fig. 25):

The Barber-Colman hardness tester is a portable hardness tester particularly adaptable to 100 per cent checking of small parts which are supposed to be modes of heat-treated bare duralumin. This "Bar-Col" indenter, made by the Barber Colman Co. of Rockford, Ill., readily spots the hardened item and from the dial gage hardness numbers are obtained which by means of the conversion chart in Fig. 26 may be converted into Brinell or Rockwell numbers.

The Webster Hardness Gage (Fig. 27):

The Webster instrument is made by R. A. Webster, Santa Monica, Calif. "It is a pliers-like tool with one fixed anvil in the form of a small cylinder. A movable hand lever operates on a plunger sliding in the head of the tool, and the plunger houses a spring-loaded indenter which has five indenting teeth. The teeth are of progressively different heights, and therefore the number of teeth which make indentations in the work forms a basis for measuring hardness since the indentations in the work depend upon the pressure to which the spring actuating the indenters is set.

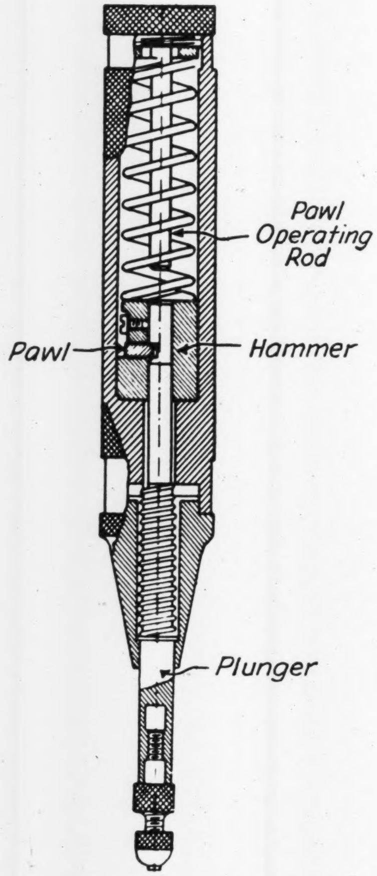

FIG. 24.—The Autopunch.

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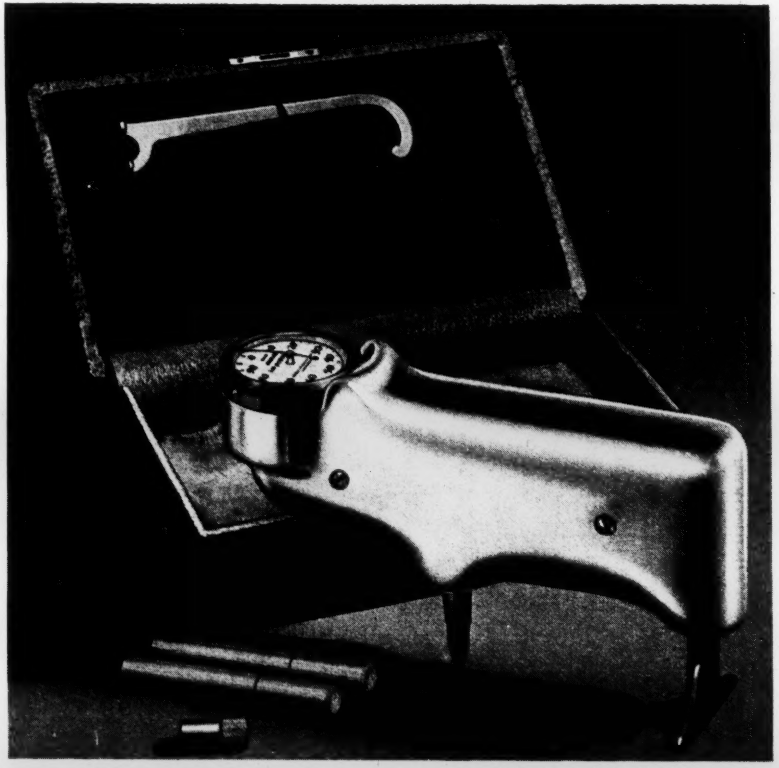
[4] P. Roudié, "Le Contrôle de la Dureté des Metaux dans L'Industrie," Dunod, Paris (1930).

[5] D. Landau, "Hardness," The Nitralloy Corp., New York, N. Y. (1943).



(*Courtesy of Barber-Colman Co.*)

FIG. 25.—The Bar-Col Impressor.

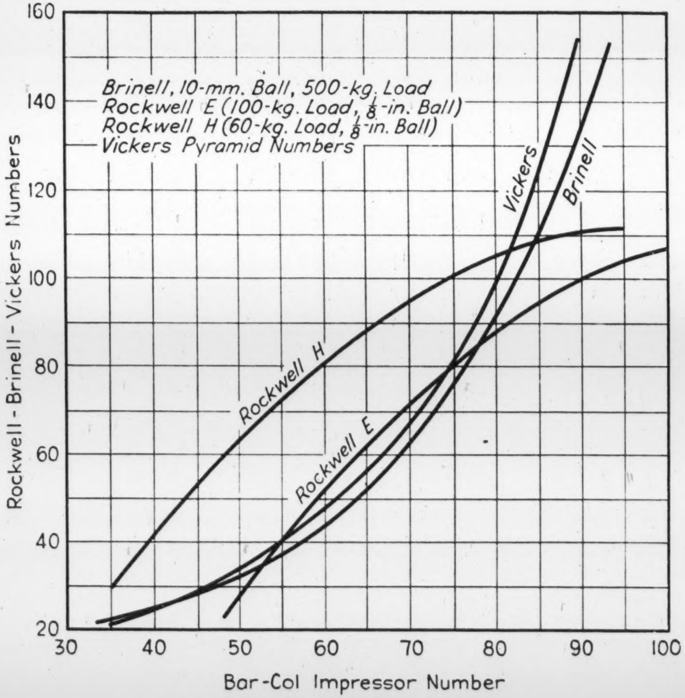
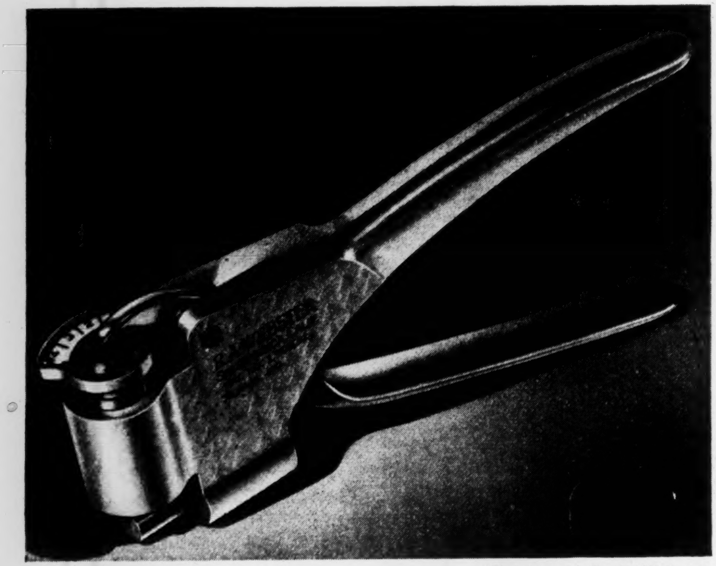


FIG. 26.—Conversion Chart for Bar-Col Impressor.



(Courtesy of R. A. Webster)

FIG. 27.—The Webster Hardness Gage. A portable instrument.

This is set by a work piece of standard hardness. It makes an excellent tool for use in airplane factories in checking the hardness of all structural work. Its place in industry can be compared to that of the "go" and "no go" gage by qualifying the work as being within the accepted standards of hardness, but without providing the hardness "number."

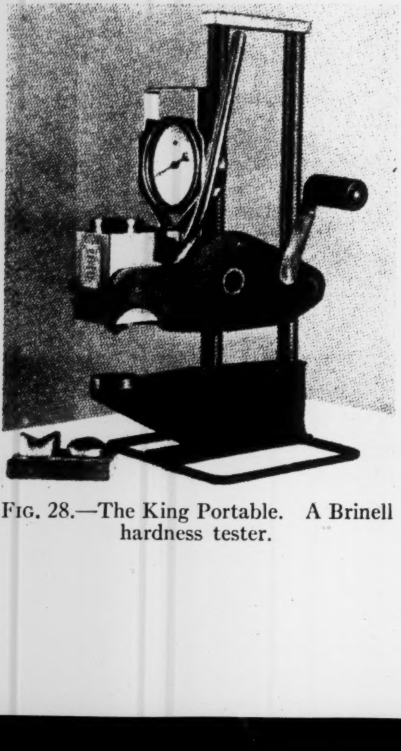


FIG. 28.—The King Portable. A Brinell hardness tester.

The King Portable Hardness Tester:

The King hardness tester is a Brinell hardness tester, one which will put a 3000-kg. load on a 10-mm. ball. The instrument is shown in Fig. 28. A distinctive feature is the removable test head, which, like the Shore scleroscope, makes it possible to test very large or heavy parts, too large or cumbersome to test in any other way. The King tester can be operated in any position—even upside down.

In this class of portable hardness testers making the standard Brinell tests up to 3000 kg. on a 10-mm. ball should be mentioned, (1) the Olsen-Mohr portable Brinell hardness tester and (2) the portable hardness tester put out by the Losenhausenwerk of Germany, which like the King tester is operated by a hand crank. Pictures of these last two portable instruments are shown elsewhere.[6]

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[6] S. R. Williams, "Hardness and Hardness Measurements," Am. Soc. Metals, pp. 357 and 359 (1942).

MAGNETIC METHODS

The practical metallurgist looks askance at any method for measuring hardness other than the old-line methods which have just been described. He has good reasons for doing so because he knows and everyone else who has ever worked with the best of these instruments knows that they are wonderful devices for checking on the uniformity of quality of material being used and for determining suitability of materials for a definite purpose.

But what is the user of an indentation method measuring? He simply does not know.

The physicist, so far as is possible, seeks to define the thing he is going to measure and then proceeds to measure it in those fundamental units of length, mass, and time. So far we have not been able to do this for hardness, as, for instance, we have done for friction, Young's modulus of stretch, viscosity, etc.

Until such time as we are able to define hardness it has seemed to the author of some importance to study correlations between well-known physical phenomena and those numbers derived from the use of so-called hardness testers. It is such a procedure which we have generally followed in setting up means for measuring physical constants. For example, when the photoelectric effect was first discovered, we did not know the laws governing its behavior; but when we knew the correlations between the output of a photoelectric cell and light intensities, then we turned around and used the photoelectric cell to measure light intensities. The more information we get about a certain subject the more intelligently can we proceed to discuss and analyze it.

For about forty years the author has had a great deal of fun playing with magnetic phenomena, particularly with magnetostriction. Not only in this special field, but also in the more general field of magnetic induction he was constantly beset by the fact that, in general, the changes in length of steel rods due to longitudinal magnetic fields and also their magnetic inductions were not so large when the numbers obtained by a Rockwell tester were high. Thus one was continually asking the question, "What is hardness?" The more one looked into the matter the more one became aware of his profound ignorance.

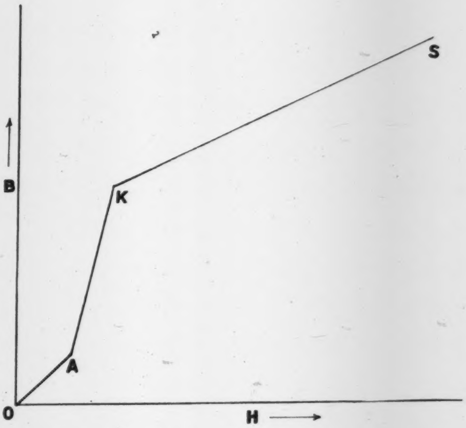

FIG. 29.—The Three Phases to a *B-H* Curve.

Space forbids going into details concerning the experiments of the two Comptons, of Barnett, of Richardson, of Chattock and Bates, and others, whereby we have come to the point of view that in a ferromagnetic body there are elementary magnets (magnetons), which are negative electrons spinning on their axes. When a bar of steel is magnetized longitudinally, it is believed that the magnetons swing around so that at the point of magnetic saturation the magnetic axes of the magnetons are more or less aligned parallel to the field impressed upon them.

In an ordinary induction curve, that is, a curve showing the relation between

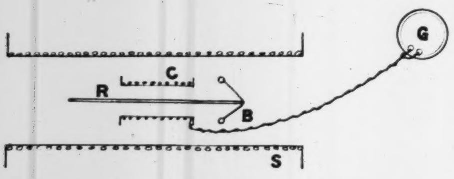

FIG. 30.—Apparatus for Comparing Intensity of Magnetization with Rockwell Hardness Numbers.

B is a rubber band for shooting the rod *R* out of the coils *C* and *S*.

the magnetizing force and the magnetic induction, there are three parts to the curve as shown in Fig. 29. In the portion *A-K*, where *B* rises most rapidly, it is believed that in this region the magnetons are aligning themselves most rapidly, and therefore *B* grows most rapdily in the portion *A-K*. For those steel rods for which Rockwell or Shore numbers are large, *B* does not grow, in general, to such large values as do those steel rods which have small Rockwell or Shore numbers.

The question arises as to which portion of the curve exhibits the most characteristic property of the material, that where the magnetons are turning around most rapidly or the portion above *K* where saturation is being reached.

Not only does it seem that the ease with which the magnetons turn around, but the degree to which they turn, must be a real property of the material. Further, the degree of turning must also hinge on the ease of turning and therefore the ease of turning is the most characteristic.

It has been elected, therefore, to compare the hardness numbers as found by a Rockwell tester with the intensity of magnetization at those field strengths where the magnetons are turning most rapidly, that is, at 8 to 12 oersteds. Figure 30 shows the arrangement of apparatus whereby uniformly machined rods of the same material but with different drawing temperatures were compared.

In the equation

$$B = H + 4\pi \mathcal{I}$$

the total flux through the steel rods is made up of the two parts, the field pro-

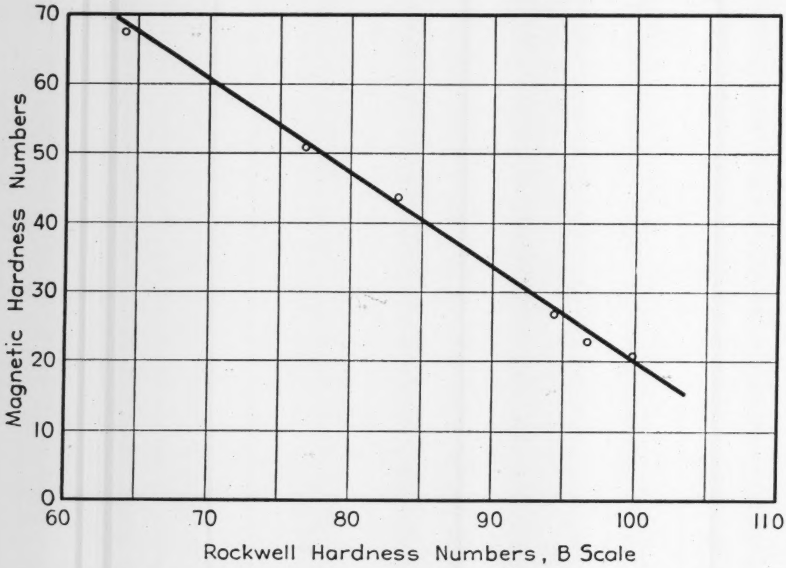

FIG. 31.—Relations Between Rockwell Hardness Numbers and Magnetic Hardness Numbers.

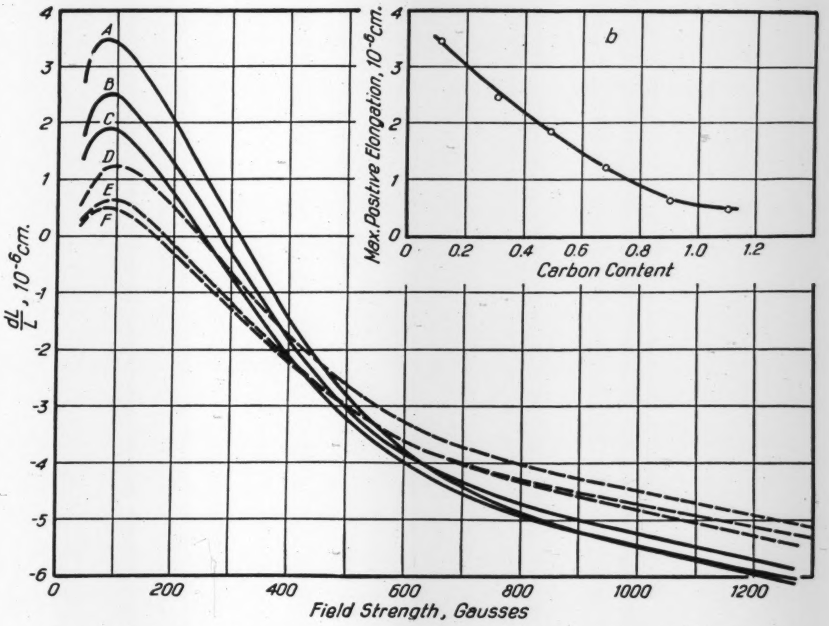

FIG. 32.—Change in Length of Steel Rods with Different Carbon Contents.

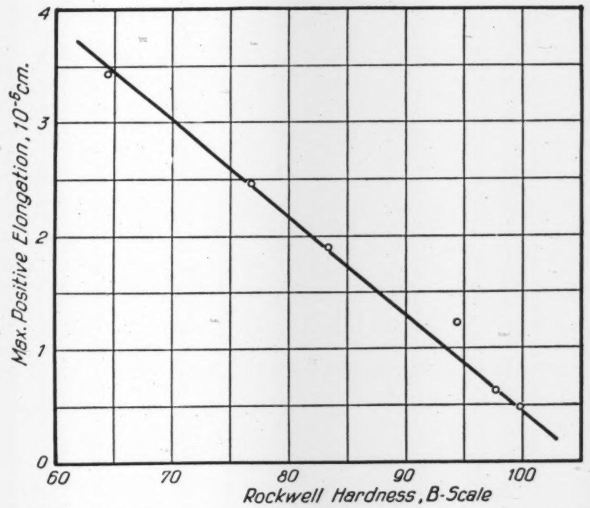

FIG. 33.—The Maximum Elongations of the Curves in Fig. 32 Plotted Against Hardness Numbers.

ducing the induction and the factor $4\pi\mathcal{I}$ which is added as the magnetons turn their lines of force to coincide with those of the magnetizing field. Consequently in Fig. 30 a steel rod R is placed within a small exploring coil C connected to a ballistic galvanometer G. This combination in turn is placed at the center of a long solenoid S. When the field of the solenoid

$$H = 4\pi nI$$

is turned on, the magnetons begin to orient themselves within the steel rod. Let this field be 8 to 12 oersteds. When the rod is suddenly jerked out of the coils, the field H remains, but that part of the total flux due to $4\pi\mathcal{I}$ goes along with the rod and as a result an emf. is set up in C and a definite deflection of the galvanometer is produced, proportional to the magnetic intensity $\mathcal{I}$. It is $\mathcal{I}$ which appears to be the real characteristic of the steel, but at such field strengths as those at which the magnetons are turning most rapidly, that is, around 8 to 12 oersteds. Fields at saturation values gave very erratic results.

Figure 31 shows the relations between the deflections of the galvanometer and the Rockwell hardness numbers. The deflections of the galvanometer are taken as the magnetic hardness numbers in the curve.

The same relations exist for rods with the same drawing temperatures and different carbon contents.

Studies were also made of steel rods hardened by cold rolling, and again the magnetic tests seem to tell a similar story as the Rockwell hardness tester.

The softer a steel rod is, the greater is the maximum elongation of that rod when magnetized longitudinally. Figure 32 shows how the rods in Fig. 31 changed their lengths as the magnetizing force increased. Figure 33 shows how the maximum elongations of these rods compare with the Rockwell hardness numbers.

If the Rockwell instrument measures a resistance to a permanent deformation or arrangement of the atoms in a sample of steel, we must think of that resistance as due to interatomic forces. By the same token these same interatomic forces operate in magnetic phenomena and to that the intermagneton forces must also be added. Do they in hardness tests? It is along such lines of investigation that we may hope to increase our knowledge of what constitutes hardness.

[See General Discussion, page 843.—ED.]

FUNDAMENTALS OF HARDNESS TESTING

By S. L. Hoyt[1]

Hardness tests, even the simple ones, become surprisingly complex when we analyze their true meanings. The most familiar of these is doubtless the Brinell test. The Brinell number is obtained as kilogram per square millimeter though by definition it is an imaginary stress. It is a sort of mean value of the resistance to indentation by the ball, but it gives neither the initial nor the ultimate resistance to penetration. In spite of its arbitrary nature, the reading serves as a valuable guide for inspection of materials, for control of composition and treatment, and it even gives an index of strength. In technical work it is but seldom that the empirical nature of the test is disturbing because most of the applications come within the natural limitations of the method. However, hardness testing, in general, is important enough to deserve most careful analysis to define those limitations and to secure sound interpretations of the results. The Brinell test is no worse off, in this respect, than other technical hardness tests and without such an analysis, hardness readings are bound to be misleading at times.

Most of the common hardness tests use the principle of resistance to penetration by a ball, cone, or pyramidal indenter. Others use the scratching principle or the rebound effect. These do not all tell the same thing about the sample and, hence, there is no common treatment for the various tests, and conversion from one scale to another must be restricted, for accuracy, to closely delimited calibrations. Another way of saying this is that there is no single property of "hardness" that is measured by these tests.

The Brinell Test

The first of the indentation tests was Brinell's which standardized on a 10-mm. ball, with a load of 3000 kg. for hard metals and 500 kg. for soft metals to keep the penetrations on the shallow side. In so far as I know, he did not fully account for his selection of the spherical area of contact by which to divide the load, but it may be assumed that he did so deliberately. The first hardness testing was on brittle materials, and when metallurgists started similar studies on the metals, they were confused by the plastic deformation and work hardening of their samples. Brinell tried various ways of securing a usable reading and was aware of the effect of the load used in the test. Ultimately he adopted the spherical area of the impression which varies in such a way with the load that the quotient (Brinell hardness number) remains fairly constant for a range of loads.[2] That must have seemed to be a good practical solution though we may note he cautiously called the quotient a hardness *number*. No doubt he knew it was a purely fictitious stress.

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[1] Battelle Memorial Institute, Columbus, Ohio.

[2] A point that is not clear here is why, knowing that the metal work hardened under the ball, did Brinell resort to the artificiality of his hardness number to obscure it.

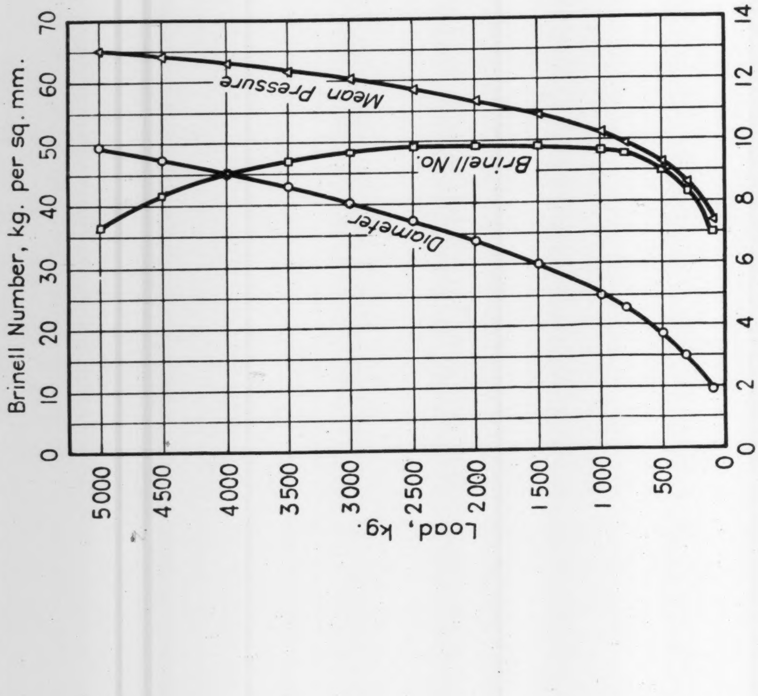

FIG. 1.—Brinell Tests of Annealed Copper.

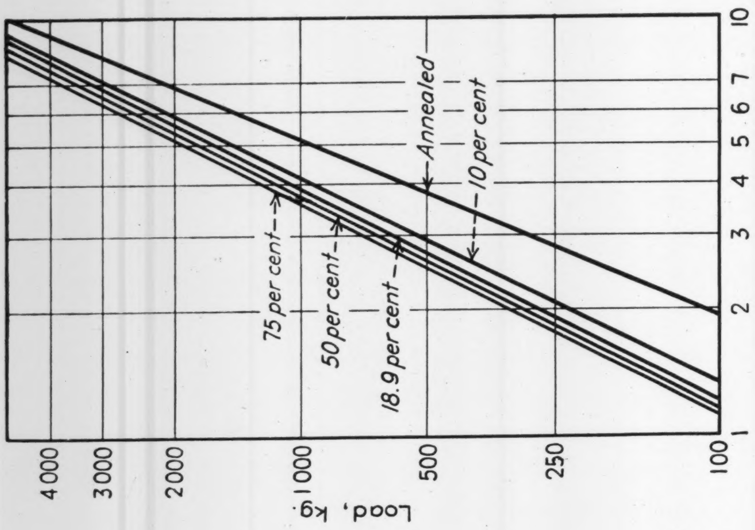

FIG. 2.—Composite Log-Log Plot of Impression Diameters of Cold-Rolled Copper.

MEYER'S ANALYSIS

An adequate analysis of the ball indentation hardness test was given by Meyer (**2, 3, 4**)[3] and has been used by students of hardness testing. Everyone is familiar with the fact that the ball is embedded deeper into the sample as the load on the ball is increased. Meyer's contribution was the finding that the rate at which the ball penetrates is a unique characteristic of the metal and that different metals respond in quite different manners. Out of this came the so-called "Meyer analysis."

Using copper as an example, a simple plot of the impression diameters for a series of loads on the ball gives a smooth curve, as in Fig. 1. Plotted on rectangular coordinate paper, this curve does not seem to have much significance. Now if we calculate Brinell numbers for the different loads (not a legitimate use of Brinell's method), a rather unusual curve develops. Far from remaining constant, as one might expect of a hardness number for pure copper, it increases rapidly at the start, to 1000 kg., and then remains constant up to 3500 kg., whereupon it falls slowly to about the initial value. One might ask which of the values represents the hardness of copper; the variation is just 40 per cent. The least that can be said of this situation is that it must lead to serious misunderstandings if we depart from Brinell's method, or compare different metals on the basis of Brinell numbers, or attempt to convert broadly from that scale to another.

The true resistance to penetration is given by the load on the ball divided by the projected area of the impression, omitting for the present any error due to elastic recovery. These values for annealed copper plot in Fig. 1 as a smooth curve, and they increase continuously with the penetration. This behavior reflects the actual increase in hardness of the annealed copper by the work-hardening effect of penetration. Meyer showed that the diameter of the impression, d, can be expressed as a simple function of the load, P, as follows:

$$P = ad^n$$

The values of P and d plot as a straight line on log-log paper as shown in Fig. 2. That this is so can be seen by converting the equation to logarithmic form:

$$\log P = \log a + n \log d$$

This represents an experimental finding but lends itself to an interesting analysis of the ball hardness test. The load and, therefore, the resistance to penetration, depends on (that is can be expressed as) the load required to produce the "initial indentation" plus a factor which evaluates the work-hardening effect as the ball continues to indent the sample. A simple procedure for approximating the initial or true hardness of the material is that of using the Meyer constant a, which is the load P for the impression diameter d of 1 mm. This can be converted, when desired, into kg. per sq. mm. Here we should note the restriction or provision that this is valid only for ball diameters, D, which are large enough to produce but very little cold work for the impression diameter of 1 mm. This seems to be satisfied for the common 10-mm. ball (**5, 6**). From the straight-line plot, the load P is read off for the impression diameter d of 1 mm. and converted into kg. per sq. mm. by dividing by 0.785. This gives the hardness of the sample effectively unaltered by penetration hardening. For annealed copper this value of P, or a, would be about 20 kg., while the hardness would be 20 ÷ 0.785 or 25 kg. per sq. mm. This value of 25 is (approximately) the hardness of annealed copper which we all talk about but never determine or use. The Brinell hardness number would be about 43.

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[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 842.

It is also simple enough to take into account the work-hardening effect of penetration, if any, since it is given by the slope of the straight-line plot, or the constant n. To illustrate this, let us first consider the case of a severely cold-worked metal which has been so effectively work hardened that it undergoes no penetration hardening. The resistance to penetration remains constant and any load on the ball is simply proportional to the projected area of the impression. In terms of the Meyer equation, P is proportional to the square of the impression diameter:

$$P = ad^2$$

This also means that the hardness is independent of the load used to determine it, since both P and the projected area of the impression (of the relation ball hardness = $P \div$ area) increase as the square of the diameter d. The quotient must remain constant.

In other cases, specifically of annealed metals which do work harden, it is found that the load increases faster than the area of the impression. This is due to work hardening of the metal during indentation, and the constant n is found to be greater than 2. If a series of determinations of P and d are plotted on log-log paper, the slope of the line is greater than the 2:1 ratio of the previous case. For all such cases the hardness readings depend on the load used in the determination as distinguished from the case of the work-hardened metal. The value of n for annealed metals may run as high as 2.5 and higher which represents a high capacity for work hardening. Figure 2 shows log-log plots for annealed and cold-rolled copper.

In an elaborate study of the Brinell test, Heyer (5) has brought out the quantitative effect of these factors in hardness testing. Using a soft steel whose a value is 66, as an example, the initial ("pretest") hardness was 85 kg. per sq. mm. or $66 \div 0.785$. For the case of $n = 2.0$, when the hardness is independent of the load used, the Brinell number was 75. For the case of $n = 2.4$ the hardness at 3000-kg. load was 160 kg. per sq. mm. while the Brinell number was 147. Thus, in these two cases though the hardness of the virgin metal, that is unaffected by the indentation process, was 85 kg. per sq. mm., the Brinell number varied from 75 to 147, depending on the value of n. The resistance to indentation at the load of 3000 kg. varied from 85 to 160. This gives a hint of a cause of some of the vagaries and irregularities often noted in hardness testing: failure to take into account the two-parameter relationship; the initial hardness, represented here approximately by a; and the effect of the indentation process, represented by n. A single number fails to tell how much of the hardness value is due to the hardness of the metal *per se* and how much to the work-hardening effect during the test. In an earlier paper (3) I gave a case which brings out this point quite clearly. A bar of cold-rolled copper had a higher Brinell number than a bar of annealed steel, yet the steel scratched the copper and in turn was not scratched by the copper. This raises the legitimate question of which was the harder of the two metals, the answer to which is given by the Meyer analysis—both. At low loads, the copper was harder, and at high loads, the mild steel, the former being more to the point, however.

CONE TESTS

With these variations of the ball test in mind, we may well ask what the situation is with other common indentation tests. The Rockwell cone and the Vickers diamond pyramid test are adaptations or refinements of the Ludwik cone test in which an indenter is used which produces geometrically similar impressions at different loads. This differs from the ball test which gives geometrically dissimilar impressions. With the cone the hardness reading does not vary with the load since the degree of indentation hardening remains constant. This advantage is utilized by the Vickers method by using loads which vary from very small to large, without affecting the scale. While gaining this advantage, however, the test suffers the restriction of determining the hardness at just one point on the hardness-penetration hardening curve and is incapable of giving the initial hardness.

More detailed information is available on the Rockwell ball type of test since Heyer has analyzed this field very thoroughly (7). The Rockwell test, whether ball or cone, utilizes the depth of the impression, but sometimes metal is forced up around the indenter (when $n =$ 2.0 to 2.25) and sometimes the impression sinks into the surface (when $n > 2.30$). This introduces an error and Heyer found that two metals of similar Brinell numbers, of 91 and 105, varied in the Rockwell test by 100 per cent, or from 33 to 66, due to this factor.

EFFECT OF RECOVERY

In nearly all tests the measurement is made on the recovered impression, whether depth or diameter. "Errors" due to recovery and to variable modulus of elasticity have been pointed out in the literature and are in the direction of giving values on the high side for the harder metals, as compared to similar determinations made on the unrecovered impressions. O'Neill (4) has described a method for carrying out tests under full load using a diamond ball. Scott and Gray (8) have shown the significance of the elastic modulus in developing correlations among hardness tests for the hard metal carbides. The Tukon test (9) can use either the recovered or unrecovered impressions by using the unique features of the design of the indenter.

TIME OF LOADING

The time of loading is important with soft and medium-hard metals. The time varies from almost instantaneous, as with the scleroscope, to 30 sec. for Brinell testing. Working with copper, I have found that at the 30-sec. point in Brinell testing the ball is still sinking into the surface. The reading at that point varied about 5 per cent from one made after the ball had reached equilibrium. With steel, Guillery (10) has reported a variation of about 8 per cent due to neglect of the time factor. In this same category comes the condition of the surface, whether clean or lubricated, as has been brought out by Hankins (11). If one is determining the Meyer constants, the load should be left on until a constant impression diameter is secured. In technical practice it is undoubtedly a proper simplification to adhere to the standardized rules for conducting the tests, but in scientific quantitative work, this unnecessary and avoidable error should be eliminated.

OTHER TESTS

Various other tests are used for hardness, such as scratch, wear or abrasion, drilling, machining, and filing. The metal is attacked in a different way and undoubtedly different properties and characteristics are brought into play so that these tests are not so easily analyzed as those of indentation. A scratch test may stress the metal locally above the cleavage point or it may simply mark a line on the surface without removing any metal. These two cases are doubtless different. It is probable that this severe deformation is responsible for the finding, that has been reported on several occasions, that the scratch test returns annealed and cold-worked metals as being the same in hardness. If the test is severe enough, whatever condition the metal may be in initially, it is always severely cold-worked metal that gives the hardness figure. (This is the same as a similar situation in the tension test. The hardness at the necked-down section is that of severely worked metal whether the test bar was annealed or cold worked initially.) Other methods mentioned remove metal as dust, filings, or chips. Except on brittle material, they first deform the metal locally. There need be little wonder that these methods fail frequently to correlate with others.

THE MEANING OF HARDNESS TESTS

The foregoing picture of hardness testing presents the results of careful and thoughtful experimental work. It is not speculation devised to explain the property, or even to explain the tests which are used to determine it, though some work has been done in the latter field (5). Perhaps the weakness of this field of investigation has been its limitation to test methods with too little consideration of the "property" itself. We still have no conception of hardness as a simple property nor do we have a precise definition. Until we can define it, we cannot set out very confidently to measure it,—so we resort to arbitrary determinations of hardness numbers. Possibly the point that confuses the issue more than any other is the alteration of the metal during the test, thus changing the very property whose measurement is sought. Thus, if we conceive of hardness as resistance to penetration, it is natural to use a penetration method, and we run into the workings of the "uncertainty" principle. When we speak of the hardness of copper, we do not ordinarily mean that, but, instead, the hardness when indented a certain amount; and the two may be quite different. In the special case of deep-drawing applications, the ordinary hardness readings are notoriously inadequate. That may go back to their inability to distinguish between initial hardness and the work-hardening effect. What a steel appears to need is a low a value and a high n value to stretch uniformly. Manganese steel (Hadfield's) and its extraordinary ability to work harden and still remain tough offers another example of a type of hardness that is missed by the usual tests. The Brinell test shows that the hardness number increases from about 200 to well over 400 when the properly quenched steel is work hardened, but this gives no clue of the extent to which work-hardening capacity is retained. In some tests of this material I found that the increase in hardness was due to the three-fold increase in a while the n value was still up to 2.15. With copper, for example, I found that when n was reduced to 2.15 on work hardening, both the a value and the ordinary hardness number had increased but a relatively small amount. In other words, Hadfield's steel work hardens quickly and considerably without losing its work-hardening capacity.

HARDNESS TESTING IN SCIENTIFIC STUDIES

In scientific studies we usually aim to establish quantitative relationships. For this the usual hardness tests should be ruled out for the reasons given above. For example, one may wish to compare copper and iron in connection with some problem, let us say, of atomic volume or structure. In this case he would undoubtedly wish to determine the property of the metal unaffected by any work-hardening effect, whether extraneous by cold working or by indentation during the test. If the determination is made at any of the higher loads, the hardness value is seriously diluted or modified by work hardening. Going to high loads, at which all initial conditions of the metal return the same figure, does not eliminate this feature. For the initial hardness the *a* value, determined with a 10-mm. ball, gives a good first approximation, and it can be improved upon by extrapolating to lower loads which correspond to still smaller impressions. Tests designed to check this point indicate that one can extrapolate well below the *a* load without increasing any important error (6). One precaution should be advanced here which relates to the error of the determinations. Several pairs of P and d values should be determined and plotted on log-log paper and these points must fall on a straight line with but very little deviation. This gives valuable evidence of internal consistency among the readings. Furthermore, I have observed that neglect of the time factor and the error introduced by the "anvil" effect give false determinations of the slope of the line (12). When these experimental conditions are satisfied, it should be possible to compare metals for hardness at small d values to eliminate penetration hardening as an important error in scientific studies.

TECHNICAL HARDNESS TESTING

Technical practice is not concerned with the niceties of the mechanics of hardness testing so long as a handy tool is provided which works. In this field we assume, with no feeling of compromise, that hardness is simply resistance to indentation. The choice of the method will depend on such factors as the hardness level, size of the part, condition of the surface, object of the determination, etc. Diamond-pointed indenters have come into use for hardness levels above 400 Brinell, not on account of theoretical advantages, but because they can stand the high pressures without distorting. The Rockwell tests are popular largely because the reading is obtained directly off a scale. Errors due to piling up or sinking in are sidestepped by comparing the reading with standard values obtained on the same metal in the same condition. This also avoids errors due to testing at different levels of indentation hardening. While this simplification is highly desirable for technical practice, it also imposes severe restrictions. We must not assume, for example, that two different metals are equally hard when they have the same hardness number, or that a cold-worked metal with twice the hardness number of an annealed metal is, therefore, twice as hard. All we can know is that a metal, when processed in a certain way, either does or does not have a required hardness number. Hence, the theoretical features, which exclude the test from being a true measure of hardness, play no part in the application of the test in technical practice when properly done.

HARDNESS CONVERSIONS

Perhaps the most difficult field in technical hardness testing, from the standpoint of fundamentals, is that of converting from one scale to another. There the factors of sinking in or piling up, indentation hardening, modulus of elasticity, etc., make it impossible to translate from one arbitrary scale to another arbitrary scale. The most that can be done is to calibrate one scale in terms of another for a certain material, such as steel,[4] cold-worked brass, etc.

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[4] See Tentative Hardness Conversion Tables for Steel (Relationship Between Diamond Pyramid Hardness, Rockwell Hardness, and Brinell Hardness) (E 48 - 43 T), 1943 Supplement to Book of A.S.T.M. Standards, Part I, p. 342.

Within the rather restricted limits of such a calibration one scale can be satisfactorily converted into another. That is done for the common tables which have been established though there is more than a suspicion that the fundamental principles have been unheeded in certain tables. Obviously, a table made for steel should not be used for hard carbides, cold-rolled copper, nickel alloys, etc. The error would be greater, if one did so, than might be suspected. Furthermore, an error would go unsuspected since there is nothing in a hardness reading to suggest the presence of error.

There is a real need for accurate conversions from one hardness scale to another and for all the important metals and alloys. While a good start has been made, there is still much more to be done and there is no more appropriate body to take this work in charge than this Society. Careful work is needed, carried out with a clear conception of the fundamentals of hardness testing, as illustrated by the work of Heyer. The tables which would ultimately be set up should define the limits of the field covered and should give the accuracy with which the conversion can be made. It would also be highly desirable in each case to give the significant Meyer constants and physical properties of the materials covered.

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GENERAL DISCUSSION

MR. F. M. HOWELL[1] (*presented in written form*).—It is interesting to note that all three of the speakers mentioned the limitations of the hardness test and emphasized the necessity of following a standardized procedure in inspection testing.

Regarding the aluminum alloys, we are frequently asked to give hardness values which can be considered as indicating definitely that the material is of the correct alloy and that it has been properly heat treated for the purpose for which it is to be used. Such values would be extremely useful, not only for users of aluminum but also for producers. If hardness limits could be established which would permit one to establish the fact that a piece of aluminum sheet had the composition, tensile properties, fatigue characteristics, formability, and resistance to corrosion expected of it, by the simple expedient of making a Brinell, Rockwell, or some other type of hardness test, much time could be saved and expense eliminated. Unfortunately, it is not quite as simple as that.

As was pointed out by Mr. Templin in 1935 before this Society,[2] and by other investigators, the Brinell hardness number of wrought aluminum alloys can be approximated fairly well by dividing the tensile strength in pounds per square inch by a constant, and that constant seems to be about 530 on the average. Actually, however, the constant for some compositions and tempers of aluminum alloys is as low as 450, while for others it is as high as 630. In general, the ratio of tensile strength to Brinell hardness is higher for materials having a relatively low yield strength, that is, a high susceptibility to cold working, and lower for those with a relatively high yield strength. These statements apply to Brinell hardness numbers obtained from tests that are made under very carefully controlled conditions. The surface of the specimen must be well prepared so that it is smooth, flat and clean, the specimen must be properly supported, the load must be applied slowly and steadily, the load must be maintained for about 30 sec. and the average diameter of the impression must be determined carefully and accurately.

Brinell hardness values can be obtained from tensile strength values. When an attempt is made to determine tensile strength from Brinell hardness, however, the results are not always so satisfactory. One reason for this is that we are generally satisfied to know the Brinell hardness within 10 or 15 points, which in the case of heat-treated aluminum alloys means about plus or minus 10 per cent. What we usually want to know about tensile strength, however, is whether the material has a tensile strength at least equal to a specified minimum value, and frequently we want to know how much it is above that minimum. The only satisfactory way to find this out is to make a tension test.

The Rockwell hardness tester is a very useful machine. It was developed primarily to provide a means for testing heat-treated alloy steels which could not be satisfactorily tested with the ordinary Brinell machine. It serves this purpose admirably. Its use has been extended to the testing of softer metals by using larger ball indenters and by reducing the magnitude of the major load. This has worked out satisfactorily for the testing of certain metals, notably cartridge brass, for which the Society has recently adopted a Standard Hardness Conversion Table for Cartridge Brass (E 33 - 42).[3] It is interesting to note, however, that this table is limited to the relationships among diamond pyramid hardness, Rockwell hardness, and Brinell hardness, and that it does not include tensile strength. The A.S.T.M. standard also emphasizes the limitations of the use of the table. It is "recommended for use in converting the results of one form of hardness test to another only when the specific test procedures and precautions outlined in the several hardness test methods" are followed. Furthermore, "these conversion values are applicable only in the case of hardness test results obtained on flat specimens of cartridge brass of sufficient thickness so as to avoid anvil effect." There are other precautionary notes in the standards which are significant, but which need not be repeated here.

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[1] Chief, Materials Testing Division, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[2] R. L. Templin, "The Hardness Testing of Light Metals and Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 283 (1935).

The Rockwell hardness tester can be used for testing aluminum alloys and it is very useful for that purpose provided it is correctly used and its limitations are appreciated. In all hardness tests, Brinell or Rockwell, it is essential that the requirements of the Society's test methods be met. Of course, as Mr. Hoyt has pointed out, for experimental work it may sometimes be desirable to depart from the standard methods. These requirements have been established only after thorough study of the tests and factors affecting the test results showed them to be necessary.

We have made large numbers of hardness tests on aluminum alloys and have studied the tests and the results obtained from them. As a result of this work we have observed some approximate relationships between Rockwell and Brinell hardness values for wrought aluminum alloys. At the present time, however, we do not feel that they are sufficiently reliable for us to present them.

It should be realized that hardness tests are made to determine hardness and that many of the conclusions drawn from the results of hardness tests are not justified, but in spite of this the hardness test not only can be but is a very useful inspection tool.

MR. H. F. MOORE.[4]—In connection with the work of the investigation of fissures in railroad rails at the University of Illinois, we have found by experience that a carefully prepared correlation diagram between Brinell hardness and Rockwell "C" hardness is not sufficiently accurate as a basis for acceptance standards. Such a diagram has been quite useful in approximating a translating of Brinell to Rockwell or *vice versa*, but when a rail customer attempted to use it within very close limits of acceptance values, it was not sufficiently accurate. I think that in any of the hardness conversion tables probable variations might well be included, and that, for special purposes it is rather unwise to use any of the hardness conversion tables which have been put out heretofore, as a basis for acceptance or rejection of material.

MR. R. L. TEMPLIN.[5]—In the hardness - tensile strength relationship data presented by Mr. Lessells the correlation was apparently quite satisfactory. While we in the Aluminum Research Laboratories have tested only a limited number of steels, yet we are unable to check some of the values presented by Mr. Lessells. The *K* values we have found show a much wider spread than he has indicated. He has made the point that there is a good relationship between hardness and tensile strength and since the fatigue strength is some simple fraction of the tensile strength, therefore, hardness is closely related to the fatigue strength of any steel. In our tests we have found ratios of fatigue to tensile strengths not ranging from the 0.4 to 0.5 mentioned by Mr. Lessells, but from less than 0.3 to 0.7. The data that he has presented apparently apply more particularly to structural steels of the low alloy type. The data which we have obtained are particularly pertinent to heat-treated high-strength alloy steels such as are used in dies and highly stressed parts of machines.

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[3] 1942 Book of A.S.T.M. Standards, Part I, p. 925.

[4] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[5] Assistant Director of Research and Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

In the same way in the case of the aluminum alloys, we find that the ratio of fatigue strength to tensile strength varies from less than 0.2 to 0.5. In so far as the relationship between hardness and tensile strength of the aluminum alloys is concerned, we obtain a simple satisfactory relationship for a commercial heat-treated wrought alloy when it is in the annealed condition; another relationship when in the solution heat-treated condition; and yet another relationship when in the fully-aged condition (solution heat treatment followed by precipitation treatment). The *K* values for these relationships, however, do not check each other and in some instances differ appreciably. Cold rolling of a heat-treated aluminum alloy after heat treatment, gives yet another *K* value. We have, therefore, concluded that there is no single satisfactory *K* value which can be accepted as defining the constant ratio of hardness to tensile strength for the aluminum alloys.

In Mr. Williams' paper he has indicated one type of hardness testing machine that is generally designated as "a rapid hardness testing machine." These machines are quite widely used and serve a very useful purpose in many commercial fields. Unfortunately, the values that these machines give for hardness are not quite the same as those obtained by following the specifications of this Society or Government specifications for the determination of Brinell hardness. Since these rapid hardness testing machines are particularly useful in routine mass inspection of war materials and because of the differences in the hardness values which they give when compared to the standard Brinell hardness test, it would appear that our Society may have been somewhat lax in not recognizing this situation and having taken proper steps to issue specifications for covering the rapid hardness tests.

MR. L. B. TUCKERMAN.[6]—Our experience with the effect of inertial forces upon Brinell and Knoop indentations makes me suspect that the differences reported by Mr. Templin between "rapid hardness testing machines" and the standard Brinell test are at least in part caused by such effects.

There is one other caution with respect to these machines. Apparently because of the rapidity of their action a given number of impressions made by them may deform or wear down the indenting tool much more than the same number of impressions in the standard tests.

We tested one of these particularly rapid testing machines some time ago making 100 tests in succession. The readings decreased progressively and the difference at the end of 100 indentations was outside of any reasonable tolerance. Examination of the indenting points under a microscope readily showed the reason in dulling of the points.

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[6] Assistant Chief, Mechanics and Sound Div., National Bureau of Standards, Washington, D. C.

MR. E. O. DIXON.[7]—In specifying hardness ranges for process control, a great deal of difficulty can be avoided if the range be specified in terms of a hardness test method practicable of application in manufacture. For instance, it is not reasonable to specify a Rockwell hardness range on heavy forgings that are heat treated, with scale on them, and have decarburization on the outside, and that weigh anywhere from ten to several hundred pounds. By the same token, as has been pointed out, it is also not practical to specify a Brinell hardness on parts that are so fragile they will not bear the pressure of a standard Brinell test without damage. So I would suggest that consideration of this sort be applied everywhere hardness ranges for manufacturing control are to be specified. Conversely, let processors protest hardness specifications couched in terms of test methods not practically applicable to the specific articles involved. By such action much unnecessary friction may be avoided with profit to all concerned.

In general, the differentiation made in several instances between what might be called scientific hardness studies and practical or technical hardness measurements for acceptance testing or for process control was very well brought out and should be kept in mind at all times in these hardness problems.

MR. EUGENE C. BINGHAM.[8]—Hardness and plasticity are alike in that both are complex properties. Plasticity is a function of two parameters, namely, yield value and mobility. We cannot define plasticity simply but we can give quantitative definitions of both yield value and mobility so it is not necessary to worry over the fact that a similar quantitative definition cannot be framed for plasticity. It is suggestive that the Meyer analysis yields *two* parameters.

In the study of plasticity it seemed that the fundamental properties involved were first, the ability to flow under shearing stress, when adequate and, second, the ability to resist flow entirely, that is, to "stay put" when the shearing stress was below the critical value. This latter may be the same as the yield point of the metallurgist, but to avoid doubt the new name "yield value" was chosen. The ability to flow may be the fluidity of a plastic material, but to save confusion again a new name was chosen, namely, "mobility."

The scleroscope brings out the elastic property of a material. Since plastic flow is a function of the time and the rebound of the ball in this test is nearly instantaneous, the amount of flow is very slight. If by having different times of rebound, one could extrapolate to zero time, it would be possible to eliminate flow altogether.

In the Brinell test the flow of the material is emphasized and not the elasticity but the conditions necessary for the measurement of yield value and mobility are not adequately met. The shearing stress is not calculable, the time is not exactly recorded, and the temperature of the flowing material is unknown. This last may be extremely important, for in the polishing of metals and marble, Beilby[9] has shown that layers of material actually flow. Although it is very contrary to our preconceived ideas, that means that even in polishing operations, very high temperatures are locally produced. These ideas as to what happens when two materials rub each other have been greatly advanced by Bowden[10] and his co-workers, temperatures being measured with thermocouples up to the melting points of the metal sliders. Why should we not seriously attempt to eliminate the elasticity and measure the yield value and mobility of metals and alloys, not bothering about the term hardness except as a general term? Bowden[11] has already shown how a hard steel ball may be used to plough through a softer metal, giving two parameters and absolute values.

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[7] Chief Metallurgical and Mechanical Engineer, Ladish Drop Forge Co., Cudahy, Wis.

[8] Professor of Research, Department of Chemistry and Metallurgy, Lafayette College, Easton, Pa.

[9] Beilby, "Aggregation and Flow of Solids," Macmillan Co., New York, N. Y.

The scratch hardness method brings in the further fundamental property of ultimate strength or cohesion. That is not to be confused with the property of flow. Fracture involves many difficulties and we may well avoid them when we can.

The cold working of a metal, like the stirring of a grease or the shaking of a colloid, changes the consistency perhaps, but the elasticity, yield value, and mobility or fluidity may be the fundamental properties along with ultimate strength that should be measured.

MR. TUCKERMAN.—In response to Mr. Bingham's remarks, I may state that some of us who have been interested in indentation tests have not forgotten the effects of temperature. We have studied the effects of temperature on indentation over such ranges as we could conveniently handle in the laboratory. In the ordinary indentation tests on metals from a range of about 100 F. down to about 60 F., the differences we found were negligible in comparison with the differences which were ascribable to other variables beyond our control. We specify certain definite limits of various test conditions within which the indentation shall be made. They are essential if the tests are to be comparable. Temperature is not one of these. Apparently for the ordinary metallic materials the temperature variation within the range of ordinary laboratory conditions is not significant.

MR. A. L. BALL.[12]—I should like to be made more acquainted with what happens to various materials when they are subjected to these so-called "hardness tests" when the materials are heated up to 600 or 800 or 1000 F. I should like to know whether anyone has done any work in that field and also what sort of apparatus was used.

MR. TUCKERMAN.—We have heard some very interesting and valuable papers describing different hardness tests and telling of the precautions needed to secure accurate results from them, but no one has discussed what the hardness was which they were measuring and only incidentally have they discussed the purpose for which they made the tests.

There was, however, an echo of the idea that there was, applicable to all materials, some accurately definable and measurable property which was hardness. I do not wish to take up time discussing the papers in detail, but I hope that I can be of some help by discussing the meaning of the word "hardness" and the reasons why hardness tests are made.

I have been interested in hardness testing for a long time. It seems to me that I can best introduce my remarks by reading something that I wrote nearly twenty years ago.

> " 'Hardness' in common parlance represents a hazily conceived conglomeration or aggregate of properties of a material, more or less related to each other. These properties include such varied things as resistance to abrasion, resistance to scratching, re-

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[10] Bowden and Ridler, *Proceedings*, Royal Soc. (London) Vol. 154A, p. 640 (1936).

[11] Bowden, Moore and Tabor, "The Ploughing and Adhesion of Sliding Metals," *Journal of Applied Physics*, Vol. 14, p. 80 (1943).

[12] Research Engineer, Carborundum Co., Niagara Falls, N. Y.

sistance to cutting, ability to cut other materials, resistance to plastic deformation, high modulus of elasticity, high yield point, high strength, absence of elastic damping, brittleness, lack of ductility and malleability, high melting temperature, magnetic retentivity, etc. This confusion under the one designation 'hardness' results from the fact that there is a rough parallelism in these properties in a large number of materials. The fact that 'hardness,' thus conceived, is a conglomeration of different, more or less unrelated properties makes it impossible to correlate any one definite, measurable property with all the current implications of hardness.

"This does not mean that under the hazy conglomeration of properties which are included in the common understanding of hardness, there are not included very important properties of the material.

"Nobody doubts that the 'hardness' of the diamond is one of its most important physical properties, nor that it is necessary to accurately control the 'hardness' of metal-cutting tools or of balance knife edges, nor that the difference between 'hard' and 'soft' glass is of great technical importance, nor that there is a great difference between 'hard' and 'soft' woods or between 'hard' candy and chocolate creams, a difference which is of considerable commercial importance.

"It does, however, mean that the properties implied by the term 'hardness' in these different cases are so heterogeneous that they cannot represent definite, accurately comparable properties of the materials in the sense that, for example, density, moduli of elasticity, specific heat, etc, represent definite and accurately comparable properties of the materials.

"The clarification needed in our ideas about hardness is therefore not an all-inclusive definition of hardness, but a careful separation of the heterogeneous group of properties now included under 'hardness' into as small a number as possible of definite, measurable physical properties which shall define all the technically valuable physical properties now included under that name.

"In attempting an analysis of the technically valuable physical properties of materials which are included under the general term 'hardness,' it will be necessary to keep clearly in mind two essentially different purposes for which hardness tests are made. In the first, the test although a 'hardness' test, is not primarily to determine 'hardness.' It is not necessary nor important, although in some cases it may be desirable that the test determine whether the material is 'hard' enough for the use to which it is to be put. Its purpose is solely to ascertain by a convenient nondestructive test whether the material is in the physical state which has, by other (in general destructive) tests, been shown to be suitable. Any other sufficiently sensitive, convenient, and reliable nondestructive physical test would serve the purpose as well.

"In the second, the 'hardness' of the material, or more specifically some of the special properties which are ordinarily loosely grouped together under the term 'hardness,' are an essential factor in the usefulness of the material. Here also it is possible to use a hardness test merely as a check on the uniformity of preparation of the material, but such a limitation of its use deprives it of a large part of its potential value as a criterion of the suitability of the material. Unfortunately our knowledge of hardness is so fragmentary that in most cases we are temporarily forced to accept this limitation even where 'hardness' is essential to the purpose for which the material is intended."

When you make a Brinell test on an automobile axle you are not at all really interested in the question whether the indentation is large or small. You want to know whether the axle, which you presumably know has the necessary chemical composition, has been properly heat treated so that it is strong enough to stand up under the loads it must bear. You have learned that if an axle made of a metal having a chemical composition lying within certain limits, having been given certain mechanical and heat treatment, at the end of that treatment has a Brinell hardness lying within certain limits, that is, if a certain ball pressed down on it under a certain load produces an indentation of a certain size, it is a satisfactory automobile axle. It is not hardness you are interested in. You are using the Brinell test, this so-called measure of hardness, as a measure of the satisfactory control of the uniformity of a technical process. Since that is your purpose it does not matter what you are measuring so long as you have a reliable measure of the uniformity of your technical process. Whether the Brinell number of your material is 400 or 500, whether the Meyer's number is 2.1 or 2.5 in reality means nothing to you. You want to know whether the axle you produce or which you buy is suitable for your use. If a Brinell, a Vickers, a Rockwell, a scleroscope or any other suitable nondestructive test insures that the particular piece of metal put through the processes you know it has been put through is suitable for your use, the test has served your purpose.

I have said many times that we ought to know much more about the various complex phenomena involved in the various hardness tests. We ought to study them in an effort to find out just what combination of definite definable properties of the material are involved in each of them.

Even more, we ought to study and are studying the much simpler problem of the plastic deformation of materials under uniform biaxial stress. Until we have a fuller understanding of this much simpler problem we cannot hope for a full understanding of indentation hardness tests. We should not, however, study them in an effort to find out whether any given test can measure "true hardness." What is true hardness any way? If there is such a thing I should like to know what it is, but I do not believe there is any property of a material which deserves that name. I think of all the indentation tests and try to think of true hardness. I can think about the stress and deformation which is produced in a material under an indenting ball. That is complex enough without trying to think of true hardness. You put a specimen under a pyramid indenter and you have an even more complex combination of stress and deformation. What relation is there between these very complex conditions which exist in the material and its true hardness? I do not know, and I fail utterly to find any significance in the thought.

We ought to study these complex phenomena until we understand them more thoroughly, but let us forget about them, for the moment, when we are talking about the usual practical hardness tests. In 99 per cent of all the hardness tests I have made, seen made, heard of or thought of it is not hardness we are interested in. We are interested in knowing whether a certain material put through certain processes is suitable for a given use. We compare the result of an indentation test, a scratch test, a rebound test, or some other sort of a test, with the success of the material in service. That is all we are interested in from a practical standpoint. That leaves us with enough problems without worrying our heads about true hardness. Mr. Williams showed a photograph of the Knoop indenter. The time we have spent in the past few years at the National Bureau of Standards in making sure that Knoop indenters shall be produced meeting our specifications within our tolerances, which we think are made as reasonable as possible, that they shall have no nicks or other defects which prevent their producing clean indentations, that the machine which the Wilson Mechanical Instrument Co., Inc. is manufacturing will apply the loads it says it does without excessive error, that the added inertial loads caused by the lifting of the beam by the specimen shall be negligibly small—the days and hours we have spent in ensuring these things in order that the Knoop indenter shall realize to the full its possibilities as an accurate and reliable tool are witness of that.

The accuracy and reliability of this instrument are important, not for the purpose of determining true hardness, but because it adds a new and valuable tool for measuring the suitability of materials for widely different uses where other instruments cannot be used.

It seems to me that it is this ghost of "true hardness" which will not be laid, but still walks abroad, which leads to the ever insistent demand for "standard" conversion tables between the various hardness scales. Recently we have even been requested to furnish a standard conversion table between the Brinell and Mohs scales.

In the National Bureau of Standards we have been engaged for a number of years on very careful tests to see how closely we could correlate one series of hardness tests with another series of hardness tests, and we have published several papers on the subject. The answer so far as we see it is this: each of the hardness tests measures a different combination of properties of the materials. A 3000-kg. Brinell test does not test the same combination of properties of the material as a 500-kg. Brinell; a Rockwell "B" tests still a different combination. If a wholly definite relation between a Rockwell "C" scale and a Brinell scale or between a Vickers scale and a scleroscope scale is found in a series of tests it can only be because the tests cover a series of materials varying only in a single parameter.

I want to make that clear. If I take steels of the same chemical composition and put them through a series of heat treatments, which consist of a quench from a given temperature and then a drawing at a series of temperatures—I wish to emphasize, the chemical composition is the same, the quenching temperature is the same and the only variable is the drawing temperature—then I feel very very sure that I can draw a perfectly smooth curve. One coordinate represents perhaps Rockwell or Vickers, and the other perhaps Vickers or Rockwell or some other indentation number. If I take that same steel and give it a different quenching temperature and give it a different series of drawing temperatures I will not get that curve but I will get something like a scatter band and the more variables I change the wider that band gets.

We found in a series of a considerable number of materials variations between the various Rockwell scales and the Brinell scale of the order of ±10 per cent within that band. If we confined ourselves to a series of materials varying by one single parameter we could set up a beautiful empirical relationship, but the moment we introduced other variables our best results would wabble every which way, consequently these conversion tables have to be looked at as only approximations.

Certain conversion tables were cited for brasses. If the brass is all of the same chemical composition, all heat treated the same way, and cold worked the same way it is a reasonably reliable conversion table. If not, only tests covering the whole range of variation in these different parameters can determine how wide its scatter band is.

The trouble with so-called standard conversion tables is this. A man submits a material for test under a specification which prescribes a certain Brinell number. We cannot make a Brinell test on it. It is too small or too thin or otherwise unsuited for that test. If we make a Vickers or a Rockwell or a Knoop test on it and convert the result into

Brinell by a standard conversion table it will be all right if the result comes within the specified limits. But if it is too high or too low, oh what a squawk! You can explain all about scatter bands but that does not help the situation.

If any so-called standard conversion table supposedly applicable to any wide variety of materials is accepted there should be full recognition of that scatter and a suitable widening of tolerance limits to cover it, provided they can be safely widened. I hate to get into arguments with a man who has specified a given hardness test on a material or specimen on which that test cannot be made. Time after time he will say "all right go ahead and use your conversion table" and later come back and raise a row, because the results were too low or too high. The real answer is not a conversion table but proper specifications. Specifications should only specify hardness tests which can be made on the material or specimen to be tested.

It seems to me that one of the reasons why the ghost of true hardness so persistently walks abroad is the narrowness of much of our outlook on hardness tests. By far the greater number of these tests are indentation tests and we forget the other implications of the word hardness. The papers presented here and the following discussions have been practically confined to indentation tests. It is with indentation tests that I have been chiefly concerned. For that reason I have always welcomed an opportunity to think of other types of hardness tests.

I was especially pleased when in a discussion of hardness my friend Theodore C. Byerly called my attention to a paper[13] discussing the hardness of the grit in chickens' gizzards. A chicken can live without grit in its gizzard and as experiments have shown can even live and thrive after its gizzard has been removed, even as a man can live with no stomach. However, that is only possible if it is supplied with a specially compounded finely ground feed. If the ordinary chicken is to live and thrive it must use its gizzard as a grinding mill and must be supplied with suitable grindstones for it. To determine what were suitable grindstones for chicken gizzards, Waite fed chickens various kinds of grit, collected their droppings and after a suitable

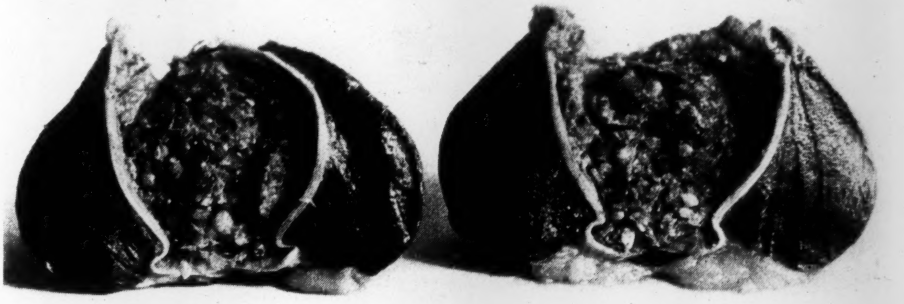

FIG. 1.—Gizzards Opened, Contents Undisturbed 48 hr. After Feeding Granite (Left), Limestone (Right).

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[13] Roy H. Waite, "Miscellaneous Studies on Poultry Grit," *Bulletin No. 377*, Maryland Experiment Station, June, 1935.

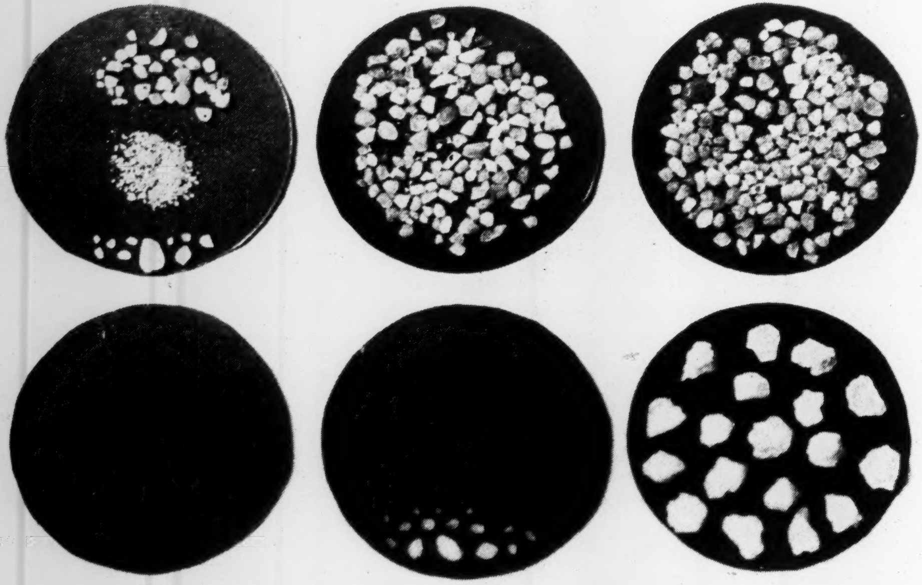

FIG. 2. Sieve Analysis of Gizzard Contents, Limestone Grit.

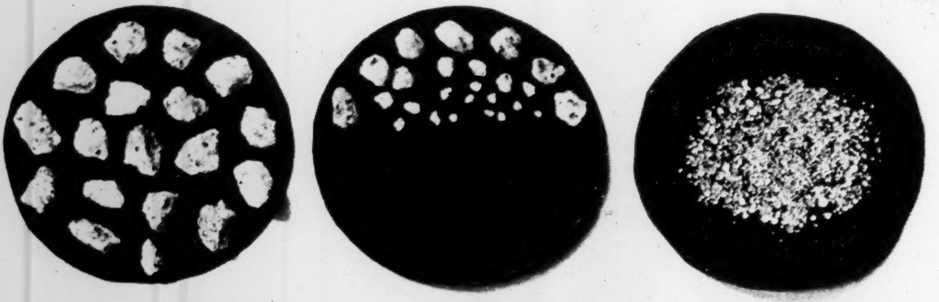

FIG. 3.—Sieve Analysis of Gizzard Contents, Granite Grit.

time had elapsed killed them, opened their gizzards (accompanying Fig. 1) and subjected droppings and gizzard contents to a sieve analysis. A comparison of the accompanying Figs. 2 and 3 will make it clear that some chicken grits may be more suitable grindstones than others. Collecting droppings, killing chickens, opening their gizzards, and analyzing their contents is tedious and expensive, so Waite sought a simpler test which could be used as a routine test. I quote:

"So much emphasis of late has been placed on hardness in grit that the writer set about constructing a mechanical device by which the various forms of grit may be subjected to a more or less uniform test. The standard test for hardness in minerals is a rubbing or scratching test, the materials being rated according to their ability to abrade or indent other minerals. The Mohs scale of hardness is the one usually used. In this scale talc is rated 1, gypsum 2, calcite 3, fluorite 4, apatite 5, orthoclase 6, quartz 7, topaz 8, corundum 9, and diamond 10.

"In handling and examining grit such as is used for poultry feeding it is readily apparent that hardness test ratings by abrasion tests do not tell the whole story. There is a shattering effect that is not taken into consideration in these tests. The proper mechanical test for grit is a machine that imitates in so far as possible the squeezing and mixing action of the gizzard, in other words, a 'mechanical gizzard.'

"The machine (shown in the accompanying Fig. 4) was recently designed and built by the writer. Two crank shafts were shaped from ½-in. cold-rolled shafting in such a manner that when revolved they would throw in opposite directions. One shaft is turned by a handle shaped from a continuation of the same material. The other is rocked by a connecting rod attached to the first. Between the two is the 'muscular part of the gizzard' made of 3-in. belting. Slots are cut in the belting in such a way as to render it capable of being folded through itself. Two coiled springs taken from the end of an ordinary bed spring connect the belting at each end of the crankshaft. These take up slack and provide a more or less uniform 'pull.' The 'lining of the gizzard'

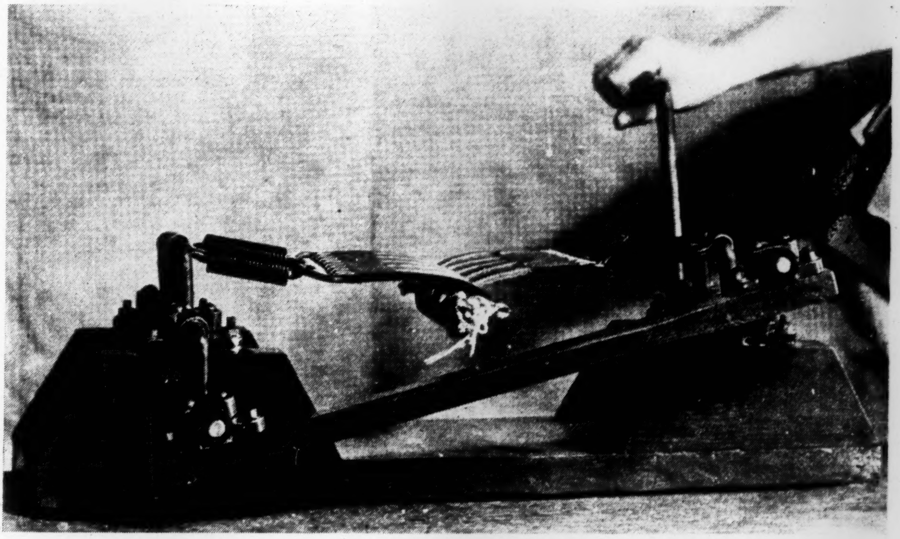

FIG. 4.—"Mechanical Gizzard" for Testing Hardness of Grit.

is made of bed ticking sewed into the form of a cylinder, and tied at both ends to retain the material to be tested. The weighted amount of sifted material is placed in this container just a little short of being packed. The container is then placed in the loop of belting. The crank is turned to make 100 squeezes, after which the material is emptied into a nest of 3 sieves, Nos. 10, 20, and 40, and shaken. The material collected on the various sieve surfaces is weighed and percentages calculated. The crank must be turned with a uniform motion to make the results comparable. In addition to squeezing, the machine produces a mixing action as the belting falls to the floor of the machine with a jarring motion with each 'throw' of the crankshaft."

TABLE I.—MATERIAL TESTED BY SQUEEZING MACHINE, ARRANGED IN ORDER OF HARDNESS.

Kind of Grit	Source of Material	Number of Tests	Amount of Material Tested, g.	Percentage of Material Passing Sieve After Squeezing		
				No. 10 Sieve	No. 20 Sieve	No. 40 Sieve
Pebble grit	Baltimore, Md.	1st	25	2.48	1.52	0.76
		2nd	24.38	1.07	0.62	0.37
Blue limestone	Morgantown, W. Va.	1st	25	7.26	5.10	2.74
		2nd	23.185	5.48	3.80	1.92
Branch gravel	College Park, Md.	1st	25	9.30	5.80	3.12
		2nd	22.675	6.24	3.84	2.03
Crushed oyster shell	Baltimore, Md.	1st	25	16.62	10.58	5.54
		2nd	20.845	16.17	10.29	5.52
Granite	Baltimore, Md.	1st	25	18.12	14.52	9.28
		2nd	20.47	11.18	8.50	5.72
Limestone (calcite)	Texas, Md.	1st	25	31.00	21.56	12.56
		2nd	17.25	22.43	16.06	10.38
Granite	Warren, N. H.	1st	25	31.06	24.36	13.14
		2nd	17.235	27.85	20.37	12.04
Limestone (marble)	Cockeysville, Md.	1st	25	37.00	32.16	19.16
		2nd	15.75	27.81	23.08	14.41
Granite	Lithonia, Ga.	1st	25	39.04	29.40	14.98
		2nd	15.24	27.40	18.96	9.68

As a result of these tests we have the chicken gizzard scale of hardness shown in the accompanying Table I. I was interested to see that, on this scale of hardness, oyster shell was harder than some limestones and granites.

You might think that this research was a nonsensical waste of time. It is not. In order that the poultry raisers of this country may raise over 250 million broilers and supply over 5 billion dozen eggs, with a market value of well over a billion dollars, for the people of the United States to consume each year, they must buy tons upon tons of chicken grit. My friend remarked "they are turning Stone Mountain into chicken grit." Incidentally, I learned on inquiring that it was not the Stone Mountain of Gutzen Borglum fame but another Stone Mountain near it which furnished so much chicken grit. It is important that our poultry raisers know what sort of hardness chickens need in the grit in their gizzards. This hardness scale has been worth many thousands of dollars to them in giving them the answer.

In conclusion I wish to repeat: "The fact that hardness thus conceived, is a conglomeration of different, more or less related properties makes it impossible to correlate any one definite, measurable property with all the current implications of hardness." There is no such thing as true hardness.

MR. S. R. WILLIAMS[14] (*Author's closure, by letter*).—Mr. Templin's suggestion that specifications be issued for the "rapid hardness testing machines" is a good one, provided these quick tests can be standardized. I believe there is some feeling that in some of these portable plier types the indenters broke down and failed to give consistent results.

"Hardness, aside from its practical importance, is one of the most remarkable properties of solid matter. This is shown by the difficulties which have been encountered in the endeavor to arrive at an accurate interpretation of it. Indeed, the attempts to solve questions relating to hardness are of very great variety, and are exceptionally large in number, and they have in measure led to some interesting results; but the subject in its broader bearings has not been attacked with success, nor has a rigorous definition of hardness been established."[15] This quotation was written in 1891. Not much can be added to that statement in 1944, over 50 years later.

It might clarify our thinking to say that there are two classes of people interested in hardness: First, those who are taking present methods for measuring resistance to penetration and seeking to analyze their results in the hope of finding out what hardness is; second, those who, breaking with traditional methods for measuring so-called hardness, are seeking a new and more fundamental way for doing what present-day hardness tests do.

So-called hardness tests do two things and do it astonishingly well: (1) determine the suitability of a material for a definite purpose, and (2) maintain standards of requirements.

However, it is a fair question to raise: Would it not be better to base such measurements on some fundamental and definable property rather than on some complex or undefinable property whose parameters are unknown? In other words those in the second class have a legitimate right to their researches as do those in the first class of investigators. A member of the first class recently suggested that the approach made by the second class "muddies the water to such an extent that a period of stagnation will be required to restore visibility." One wonders how visibility can be restored in an opaque medium.

The most constructive discussion we have had in this symposium is by Mr. Bingham who belongs to the second class. As he points out, hardness is a complex property whose parameters we do not know. The analogy which he draws between hardness and plasticity is most helpful.

Because of the proportional relationship between hardness and the tensile strength of steel it has always seemed to the author that studies should be made on the relationship between hardness and the energy of impacts (or the load applied statically) which will produce the first detectable identation. This would be at the threshold of the yield value as Mr. Bingham has suggested. This study is now under way with the instrument described elsewhere.[16] Hertz,[17] long ago, made a similar suggestion and many of his ideas are illuminating today. His definition for hardness is, "Hardness is the limiting elastic resistance (tenacity) of a body in case of contact of one of its plane surfaces with the spherical surface of another body." In our present verbiage this would be saying that the load applied to an indenter (spherical if you wish) which will make the first distinguishable indentation measures the property of yield value which Mr. Bingham has suggested would give us all the information we now have from our hardness testers.

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[14] Fayerweather Laboratory of Physics, Amherst College, Amherst, Mass.

[15] F. Auerbach, "Absolute Hartemessung," *Annalen der Physik und Chemie*, Vol. 43, pp. 61–100 (1891).

[16] S. R. Williams, "Hardness and Hardness Measurments," Am. Soc. Metals, Fig. 176, pp. 250–252 (1942).

[17] H. Hertz, Über die Beruhrung fester elastischer Körper," *Journal für die reine und angewandte Mathematik*, Vol. 92, pp. 156–171 (1881).

Quoting from Auerbach once more, "At the outset, however, it is by no means necessary that the procedure adopted should be so simple as to be of practical utility. As a rule this will only be attained at a much later stage of the research. The chief aim at the beginning is to work forward from some theoretically perfect basis, and to so fashion the methods that the end in view may be reached with a reasonable degree of accuracy as well as certainty."

The great advantage of our present measurers of resistance to penetration is their practical utility, but their development has been from the operational viewpoint and not on the basis of a clean-cut theory.

MR. JOHN M. LESSELLS[18] (*author's closure, by letter*).—It is gratifying to have the various discussions to the Symposium papers since these together with the papers bring us up to date on the present conception of hardness.

Commenting on Mr. Templin's remarks, reference to the presented data by the author indicates that the value of K referred to the high strength alloy steels as well as the low alloy and carbon grades. The value of the ratios of fatigue strength to tensile strength was taken as 0.4 to 0.6 and this ratio has been generally confirmed by thousands of actual tests. It is appreciated that under special conditions, such as high residual stress, this ratio range may have to be extended. These exceptions are mentioned in the papers. The remarks made regarding the application of K to aluminum alloys will be useful in the application of these materials.

The points raised by Mr. Dixon are important. It is quite true that specified hardness ranges must be practicable in their application, and in general the selection of a range is based on these considerations.

Regarding Mr. Ball's remarks, the Symposium only dealt with the hardness as based on atmospheric temperatures. The effects of elevated temperatures have been discussed in the literature and reference can be made to Chapter VII of the publication by H. O'Neill included in the bibliography,[19] which forms a part of this Symposium.

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[18] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[19] See p. 813.

MEASUREMENT OF BOND BETWEEN BRICKS AND MORTAR

By J. C. Pearson[1]

Synopsis

Experience with various masonry bond tests has shown that most of them give discouragingly wild results, and that good reproducibility is attained, if at all, by artificial procedures which neither imitate the bricklayer's manipulation nor produce results anything like those obtained in practical masonry construction. An extensive search was, therefore, undertaken for better methods of test, and particularly to develop a method imitating job conditions and manipulation without involving too many uncontrolled variables.

As a laboratory test, a mechanized assembly of the old cross-brick couplet was found to give the most satisfactory results for bond in tension. Another test, developed with the aid of an experienced bricklayer, consisted in prying up bricks, laid in mortar on the top of a wall, with a lever under measured load, thus permitting calculation of the stress at rupture. Analysis of many test results from the two methods showed that the couplet test is the more reliable, and that it responds to the plasticity of the mortar somewhat more favorably than the so-called wall test. But with many different types of mortar, the mechanically assembled couplet gave results that were, on the whole, reasonably parallel to those obtained from the wall test; and the couplet test therefore furnishes a simple means of estimating bond strengths to be expected in actual brick construction.

The ability of masonry mortars to bond firmly to building units is important, because lateral strength and watertightness of walls are largely dependent on this property. While bond strength is normally much lower than compressive strength, the two are not closely related and one cannot, in general, be inferred from a knowledge of the other. It is, therefore, unfortunate that there is no simple and precise method of testing bond strength, not only for a knowledge of bond in itself, but also because the effects of certain other properties, also difficult to measure, may be reflected in the adhesive quality of a mortar.

The author's interest in the bond problem was kindled some two years ago by an assignment to find out something about the quantity and effect of air produced by certain grinding aids in a prepared masonry cement. The specification tests did not seem capable of yielding the desired information, but investigation of the effects of bond strength seemed promising. This line of attack was therefore undertaken, but attempts to measure the bond by three different methods, of which it was hoped that at least one would prove satisfactory, failed to give promise of meeting the essential requirements of simplicity and reproducibility. In view of this unexpected setback, it was decided to forget the main investigation for a while, and try to find a reasonably dependable method of measuring the bond of mortar to common brick. It is perhaps going too far to say that this search has been entirely successful, but the gist of what has been learned is presented in this paper, and this may be helpful to others interested in the subject.

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[1] Director of Research, Lehigh Portland Cement Co., Allentown, Pa.

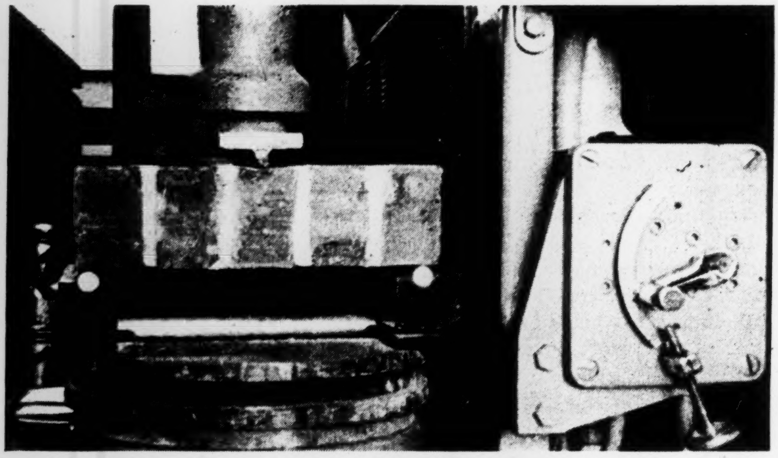

FIG. 1.—Five-Brick Beam Test.

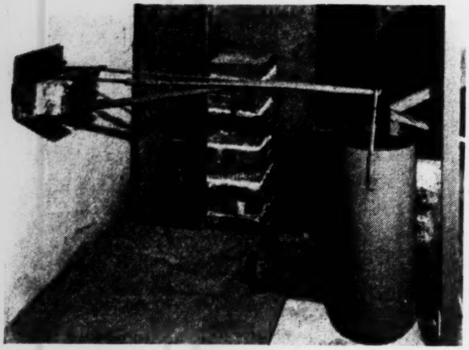

FIG. 2.—"Offset Couplet" for Cantilever Test.

It may be worth while first to describe very briefly the methods that were tried and discarded, and why they were discarded, as well as the two that were considered to have most promise and were eventually rated according to their actual performance in a large number of tests.

Figure 1 shows the familiar 5-brick beam in position in a testing machine for flexure test. The specimen is fairly well adapted for assembly by the bricklayer's methods, but it has the disadvantage of indeterminate pressure or tapping applied in the making, which has been found to be one of the major factors affecting bond strength; also, as in all hand-made couplets, the mortar squeezes out more or less unevenly and leaves an indefinite zone of little or no bond around the perimeter of the brick faces; and finally the cost of using at least three selected bricks and of laying up the beam or column is too great for the large number of tests required for a dependable average value of bond strength.

One of the early tests that had been developed in our laboratory is shown in Fig. 2. Two bricks were mortared together in such a way that the long side of one projected about $\frac{1}{4}$ in. beyond the long side of the other, and this provided for the attachment of a loading arm to the one while the other was held rigidly in a clamp anchored to the wall. This test was found to give erratic results if the couplets were made as the bricklayer would make them, and it was only by thoroughly "buttering" both bricks that reasonably reproducible results could be obtained. But these values were so high that they obviously bore little relation to the bond of mortar to bricks in a structure, and the test was discarded mainly for this reason.

In searching for a simpler test method of lower cost and one promising better control, the assembly shown in Fig. 3 was devised. The specimen was called an "end couplet," formed by sawing a brick into two 4-in. square bats and mortaring them together in a special jig. This method seemed promising because differences in surface texture of the two bonded surfaces were eliminated by the cut, and the assembly was almost entirely mechanical. A groove sawn in one of the bats permitted the attachment of a loading arm for the flexure test. Contrary to expectations, these couplets did not give reasonably uniform results even under favorable curing conditions, and variations were still greater with limited curing. Examination of the bonded surfaces after failure showed that, in general, the bond was distinctly higher in the center of the section than around the edges, and the varying width of the zones of low bond caused large variations in modulus of rupture, magnified, of course, by the small area of cross-section. The large surface-volume ratio of these end couplets also magnified the variable drying effects when stored in laboratory air, and it became apparent that bonded area and size of specimen should be larger, particularly for flexure tests.

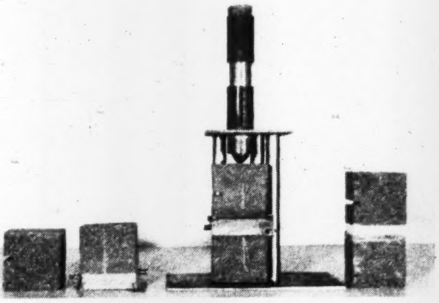

FIG. 3.—"End Couplet" Assembly.

A considerable study was made of the time-honored cross-brick couplet as a method for determining tensile bond strength. Starting with the procedure for this test as reported by Committee C-12 on Mortar for Unit Masonry and described in the ASTM BULLETIN for October, 1938,[2] indifferent results were first obtained in repeat tests, but the assembly was gradually improved by mechanical aids until it was freed from nearly all manipulation effects. In fact, it eventually proved to be the most satisfactory of all the methods studied, and it will be described in detail later.

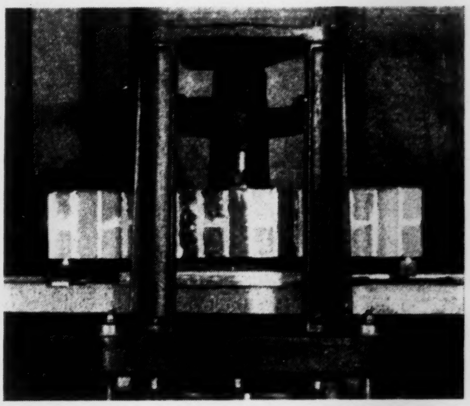

FIG. 4.—Flexure Test of 8 by 8 by 42-in. Brick Beam.

While the cross-brick couplet has given a very good account of itself, it is obvious that it does not imitate the mason's manipulation in laying bricks, and it seemed highly desirable to find some reasonably reliable method of testing the bond of bricks as laid by an experienced brick-layer. Fortunately, one of our employees in this category was assigned to the laboratory during the year, and he built a number of 8 by 8 by 42-in. piers using selected bricks and two grades of mortar, mainly to get some idea of what order of bond values might be expected. These piers were erected in a quiet room where a medium to high humidity prevailed, and remained there for 2 weeks. They were then tested in flexure as shown in Fig. 4. The deviations were considerable, particularly for the weaker mortar, but the average modulus of rupture in bond was 23 and 48 psi., respectively. These figures were assumed to be somewhat lower than they would be for ordinary wall construction under the same conditions, partly because more adjustment of the bricks is necessary in building a column, partly because a free-standing column dries out more rapidly.

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[2] "A Preliminary Consideration of Some Proposed Methods of Sampling and Testing Mortar for Unit Masonry," ASTM BULLETIN, No. 94, October, 1938, p. 40.

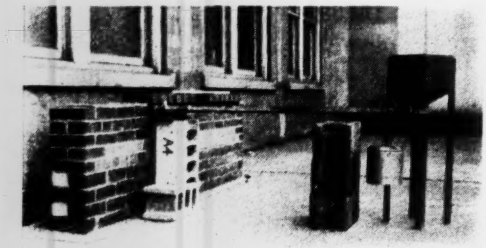

FIG. 5.—Original Outside "Wall Test."

To secure more control in a test that also involved the masonic operations, a rather novel scheme was devised, as shown in Fig. 5. A low brick wall was constructed outside the laboratory, and a course of bricks was laid header fashion on top of the wall, but spaced $\frac{1}{2}$ in. or more apart with the ends projecting about 1 in. beyond the face of the wall. This permitted the bricks to be pried up with a lever arrangement as shown in Fig. 5, and the stress at rupture to be computed from the mechanics of the system. This test proved promising, and it was further developed experimentally. It became known as the "wall test," and was eventually transferred to the laboratory where a fair approach to constant curing conditions was possible.

As a result of these exploratory operations, the modified cross-brick couplet for a laboratory test of bond in tension, and the wall test for modulus of rupture of bond,[3] carried out with the aid of an experienced bricklayer, were considered worthy of further study; and the apparatus and procedure as finally adopted for each will now be described in some detail.

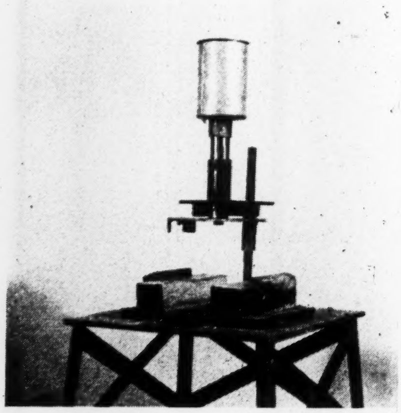

FIG. 6.—Assembly Jig for "Cross-Brick Couplet."

MODIFIED CROSS-BRICK COUPLET TEST

Figure 6 shows the couplet assembly jig. On a small table is fastened a sort of platform about $8\frac{1}{2}$ in. square and $2\frac{1}{4}$ in. high, the center portion of which is cut away to take the lower brick of the couplet. The tops and sides of the two sections of the platform are faced with sheet metal to prevent absorption of water from the mortar. Two screed strips, 6 in. long and projecting $\frac{5}{8}$ in. above the platform, are attached to the sides of the latter, parallel to the brick. The space between the screeds is filled with mortar and the excess removed, thus forming a level bed of mortar $\frac{5}{8}$ by 6 by $8\frac{1}{2}$ in. A strip of mortar $1\frac{1}{4}$ in. wide is then removed with a putty knife just inside of each screed, leaving a bed of mortar 6 in. square centered on the lower brick. The upper brick is then placed against the overhead guide, which is fastened to the bottom of a shaft that moves vertically through a rigidly supported sleeve, and is lowered gently onto the mortar bed. The upper brick is thus automatically leveled, centered, and placed at right angles to the lower brick under a superimposed weight of 20 lb. (adjusted by the shot carried in the receptacle on top of the shaft). After waiting about 2 min. for the mortar to stiffen appreciably, the couplet is carefully removed, and the joint trimmed flush with the outer faces of the bricks.

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[3] There is some question whether "modulus of rupture" is the proper term for the maximum stress when the latter is a combination of bending stress and direct tension as it is in this case. For convenience, however, the term has been used throughout the paper for the maximum stress at rupture as determined in the wall test.

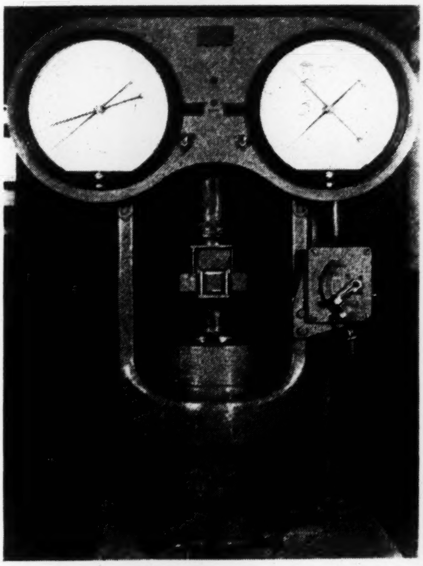

FIG. 7.—Tension Test of "Cross-Brick Couplet."

The test is made as shown in Fig. 7. Two three-legged frogs are used to support the couplet instead of the usual channel sections. Centering of the supports is done very carefully with mechanical aids. Centering the couplet in the testing machine is insured by a circle scribed on the upper frog, which indicates the proper position of the spherical block when the load is applied.

It should be noted that the procedure followed in assembly of the couplets is such as to insure failure at the top of the joint. It is the bond of the upper brick that is important, and it is this contact between the upper brick and the mortar that is under control. The bond to the lower brick may be either higher or lower than that to the upper brick, depending upon how the mortar is spread upon it. In the procedure adopted, a lower brick is used that has a low absorption and is first dampened by a short partial immersion in water, then a grout of high-early strength cement is brushed over the contact area just before assembly. Then a thin layer of mortar is trowelled hard on the grouted surface before the main bed of mortar is placed. In this manner failure will nearly always occur at the top of the joint. Not only is the bond of the upper brick more significant, but the procedure is more economical in that only one selected brick of the type under test is required for each couplet.

WALL TEST

The base for the inside wall test is a sort of altar about 10 ft. long and 38 in. wide, surmounted by four steps, with a capacity of 80 or more test bricks.

Figure 8 shows a group of bricks on the altar in process of being tested. The preferred procedure in laying the test bricks is as follows:

The altar is first prepared by wetting it down and covering it with wet burlap for a couple of days prior to setting the test bricks. Special screeds are used for laying a $\frac{5}{8}$-in. bed of mortar on each step, the one in front of the step forming an extension to the tread so that the mortar bed is 9 in. wide and projects $\frac{1}{2}$ in. beyond the ends of the brick as placed. Guide lines are set as accurately as possible at $2\frac{3}{4}$ in. above the tread, thus giving a nominal $\frac{1}{2}$-in. mortar joint when the bricks are set to the lines. Bricks are placed in position on the mortar bed and immediately pressed down by hand (without tapping) following the usual job practice. After removal of the screeds, the mortar is cut flush with the sides and back of each brick, and flush with the riser of the step. As in the case of the couplets, failure at the top of the mortar joint is desired and is forced to occur by grouting the tread before spreading the mortar. In setting up the test bricks, the four mortars usually tested at one time are rotated in position on all four steps, five test bricks being laid in each mortar on each step. This is an important precaution, for in spite of all care in laying the bricks, there are frequently considerable differences in the test results for a given mortar from one step to another. The modulus of rupture from the wall test is computed from the formula:

$$\text{Modulus of Rupture} = \frac{P}{A} + \frac{6Pe}{bd^2}$$

where:

P = lifting force applied near end of brick,

A = area of mortar bed in contact with brick ($b \times d$),

e = distance from point of application of P to the center of the mortar bed. To obtain e, the distance from point of application of P to the back end of brick is measured. If this distance is L, $e = L - d/2$.

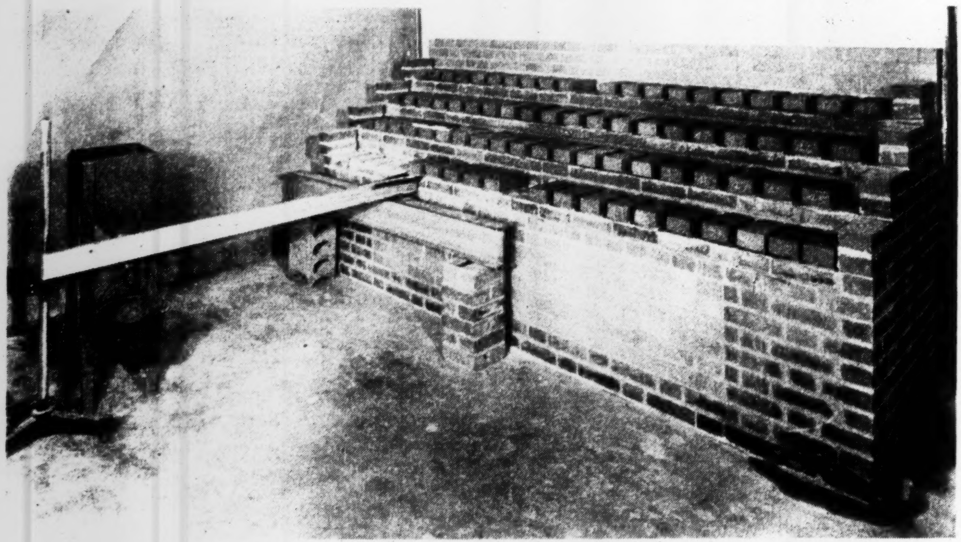

FIG. 8.—Inside "Altar" for Bond Tests of Bricks and Mortars (Wall Test).

CONTROLLED VARIABLES

Glen Gery common brick from Reading, Pa., were used in all tests reported herein. All bricks used for testing purposes were given a 1-min. test for absorption and were marked accordingly. These bricks were fairly uniform in texture but had a higher rate of absorption than was desired. The majority showed 1-min. absorptions between 35 and 60 g., but the range for any given series was held to 10 g. or less, as indicated in the tables.

TABLE I.—EARLY DETERMINATIONS OF BOND STRENGTHS FROM THE WALL TEST AND CROSS-BRICK COUPLETS.

NOTE.—All bricks used were Glen Gery common bricks with 1-min. absorption rates as indicated. Wall test bricks were set by hand on ⅝-in. mortar bed. All couplets were assembled by machine with 20 lb. load on upper brick.

Mortar	Range of Brick Absorption, g. per min.	Wall Test			Cross-Brick Couplets			Remarks on Method of Laying in Wall Test
		Modulus of Rupture of Bond, psi.	Number of Tests	Standard Deviation, per cent	Tensile Strength of Bond, psi.	Number of Tests	Standard Deviation, per cent	
A-1.........	34 to 39	73	84	26	69	70	15	Tapped from ⅝-in. bed to ½-in. joint
A-1.........	34 to 39	88	76	10	...	...	...	Tapped from ⅝-in. bed to ½-in. joint
B-1.........	40 to 44	71	85	8	55	70	14	Tapped from ⅝-in. bed to ½-in. joint
C-1.........	40 to 44	100	59	10	74	70	14	Tapped from ⅝-in. bed to ½-in. joint
A-1.........	40 to 45	75	88	17	...	...	...	Tapped from ⅝-in. bed to ½-in. joint
A-1.........	27 to 33	86	41	11	...	...	...	Tapped to uniform resistance
A-1.........	51 to 59	79	42	13	...	...	...	Tapped to uniform resistance
A-1.........	40 to 44	96	78	10	...	...	...	Tapped heavily to uniform resistance
A-1.........	45 to 54	65	20	25	60	20	12	Tapped from ⅝-in. bed to ⅜-in. joint
M...........	45 to 54	55	20	19	53	20	10	Tapped from ⅝-in. bed to ⅜-in. joint
R...........	45 to 54	71	20	19	57	20	14	Tapped from ⅝-in. bed to ⅜-in. joint
D...........	45 to 54	64	20	11	58	20	11	Tapped from ⅝-in. bed to ⅜-in. joint

When the wall test was first put into full scale operation, it had not been decided just how to set the test bricks. It was assumed that tapping them down to uniform joint thickness would be proper, but since rather large deviations were observed, several modifications were tried. Since the tapping could not well be controlled, and it seemed to have a rather marked effect on results, it was finally decided to set the test bricks by hand pressure alone, to line, as the nearest approach to normal job practice.

Usually four mortars were tested at a time, the four batches being prepared and carefully checked for consistency. Guided by the bricklayer's preferences, most of the prepared masonry cements were right when they gave a slump of 3-in. using the 6-in. slump cone. For very plastic mortars the slump for the desired consistency was 2½ in. Because the bond strength in either type of test was strongly affected by consistency, it became the established practice to recheck the slump after making five couplets, or after laying five bricks in the wall test. In general, the slump was maintained within a range of less than ½ in. throughout any given series by re-gaging the mortar occasionally. While this may have had some slight effect on strength, it was negligible[4] in comparison with the effect of stiffening.

For both types of test it was arbitrarily decided that a week's damp curing was a conservative imitation of the average curing of mortar in a building, which, of course, may be anything. This was accomplished by storing the couplets 7 days in the damp room, and by covering the wall test bricks with wet burlap and tying a single piece of Sisalkraft paper over the entire altar. Most of this work was done during the winter when the average temperature conditions were about the same from one 7-day period to another.

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[4] If re-tempering affects the bond strength materially, it should cause the average of the first ten couplets of a set to be higher or lower than the average of the second ten, in the long run. The grand average difference from all the sets listed in Tables II and III is less than 2 per cent, in favor of the first ten.

TABLE II.—COMPARATIVE TEST DATA ON BOND STRENGTH FROM WALL TEST AND CROSS-BRICK COUPLET TEST

Note.—All bricks used were Glen Gery common bricks having absorption rates in the range 45 to 54 g. per min. Wall test bricks were set by hand on ⅝ in. mortar bed to give a ½ in. joint. All couplets were made by machine with 20-lb. load on upper brick.

Mortar	Wall Test			Cross-Brick Couplets		
	Modulus of Rupture of Bond, psi. ($P_s = 0.90$)	Number of Tests	Standard Deviation, per cent	Tensile Strength of Bond, psi. ($P_s = 0.90$)	Number of Tests	Standard Deviation, per cent
D	44.1 ± 3.2	20	18.4	57.8 ± 2.5	20	10.7
E	33.9 ± 1.9	20	14.3			
F	24.4 ± 3.1	20	32.2	43.4 ± 1.4	20	8.3
G	57.5 ± 3.1	19	13.3	73.0 ± 3.6	20	12.4
H	41.3 ± 2.3	19	13.5			
I	35.1 ± 3.1	19	21.9	61.6 ± 2.8	20	11.5
J	41.5 ± 2.9	20	17.6			
K	57.2 ± 3.3	20	14.5			
L	35.8 ± 2.0	19	13.7			
M	32.4 ± 2.2	20	16.9	53.3 ± 2.2	20	10.2
N	35.2 ± 2.3	20	16.8			
O	48.2 ± 2.8	20	14.4	62.1 ± 2.7	20	10.8
P	31.6 ± 2.2	20	17.3			
A-2	42.0 ± 3.0	20	17.8	69.3 ± 3.5	20	12.6
A-3	34.5 ± 2.7	20	19.7	63.7 ± 3.6	20	14.1
A-4	37.5 ± 3.2	19	20.7	69.6 ± 3.4	20	12.2
B-1	40.4 ± 2.5	20	15.6	56.5 ± 2.3	20	10.4
C-1	55.0 ± 3.7	20	16.8	77.8 ± 4.3	20	14.0
Q	34.2 ± 2.3	20	17.2	65.7 ± 2.6	20	10.1
R	41.7 ± 2.4	20	14.3	56.8 ± 3.2	20	14.1
S	44.4 ± 3.4	19	18.8			
B-2	54.2 ± 4.9	20	22.5			
C-2	67.7 ± 4.6	20	17.0			

RESULTS OF TESTS

The results of some of the tests made prior to the decision to set the bricks without tapping are given in Table I. The first five sets were tapped as required to set the bricks down ⅛ in. into the mortar bed. Note that the standard deviations in these sets varied from 8 to 26 per cent, and that the moduli of rupture were higher than the tensile strengths from the machine made couplets. The next three sets were made according to the bricklayer's judgment of tapping the bricks down to uniform resistance. The standard deviations of these sets were not unsatisfactory, and the tests show somewhat lower bond strength for bricks of higher absorption rate, also somewhat higher bond strengths from heavier tapping. The last four sets were made at one time and show that heavier tapping to line than in the first five sets again failed to give good uniformity.

Table II gives wall test results obtained from a considerable number of different mortars when all bricks were set ⅛ in. into the mortar by hand pressure alone. The tensile bond strengths from couplets are also given for the majority of these mortars. The most striking feature of these results is the low order of modulus of rupture values as compared with those in Table I. Direct comparisons are possible only with mortars B-1, C-1, D, M, and R, but it is seen that in all cases the tensile bond strength now exceeds the modulus of rupture. The effect of tapping the bricks is, therefore, to increase the bond strength from 50 to 100 per cent, which suggests that the contact between mortar and brick is much improved by impact or by the vibration resulting from impact.

The standard deviations of the tensile strengths recorded in Table II are seen to be, in practically all cases, between 10 and 15 per cent of the average values, whereas, the standard deviations of the modulus of rupture are mostly within the range 15 to 20 per cent (some higher). Therefore, if one desires to know how many tests of each kind are required, let us say, to obtain an average that would be expected to be within 10 per cent of the average value from a large number of tests in 90 per cent of the tests made

($P_s = 0.90$) it would be conservative to assume a standard deviation of 15 per cent for the couplet tests and 25 per cent for the wall tests. According to methods given by Crum and Leavitt,[5] the number of couplet tests required for these limits of accuracy would be six and the number of wall tests seventeen. In the majority of tests reported in the tables herewith, the number of companion specimens was 20, although larger numbers were used in several of the earlier series. The plus or minus quantities following the modulus of rupture values and the tensile strengths in Tables II and III indicate the probable ranges within which the averages from a large number of tests would be expected to fall within these same limits of accuracy.[6] It is to be noted that these ranges are based on the actual standard deviations computed from the limited number of tests made in each individual case.

TABLE III.—REPEAT TESTS ON MORTARS UNDER ADOPTED PROCEDURE.

See NOTE under heading of Table II

Mortar	Range of Brick Absorption, g. per min.	Wall Test			Cross-Brick Couplets		
		Modulus of Rupture of Bond, psi. ($P_s = 0.90$)	Number of Tests	Standard Deviation, per cent	Tensile Strength of Bond, psi. ($P_s = 0.90$)	Number of Tests	Standard Deviation, per cent
A-1.....	45 to 54	31.2±3.3	20	26.7	59.5±2.5	20	12.3
		36.4±4.1	20	28.5	67.4±2.9	20	10.9
		35.1±3.4	20	24.3	67.9±3.3	20	12.3
		33.5±2.8	20	20.9	69.1±2.1	70	14.8
B-1.....	45 to 54	....	......	......	56.5±2.3	20	10.4
					54.9±1.5	70	13.8
C-1.....	45 to 54	....	......	......	74.5±2.0	70	13.6
					77.8±4.3	20	14.0

It is very much to be regretted that more repetitions of sets were not made with one or more selected mortars. Only the data in Table III were available for repeat sets in which all conditions were alike, so far as known. It may be noted that the average value for the first set of couplets is somewhat outside the expected range.

CONCLUDING REMARKS

The magnitude of the deviations in the bond test results reported in this study is evidence of the presence of uncontrolled variables of considerable importance. As a reminder of the presence of unknown variables, it has been frequently noted that bricks having the same absorption in the 1-min. test do not always produce uniform stiffening rate of a given mortar. It is known, however, that the consistency of the mortar must be closely controlled. As evidence of this, a certain mortar was gaged to a $3\frac{1}{4}$-in. slump at the start of making a set of couplets and when the first ten were completed the slump had fallen to 3 in. With the same mortar, another set of ten couplets was made, starting with a slump of $2\frac{3}{4}$ in. and ending at $2\frac{3}{8}$ in. The average tensile strength for the first ten was 68 psi.. for the second ten 44 psi. This experience indicates that the slump should be maintained within $\frac{1}{4}$ in. of the desired value if it is not to have considerable effect on the bond strength.

In the wall test, the consistency is equally important as in the couplet test, and the pressure factor will vary with wetness and plasticity, with any inaccuracy in the setting of the lines, with varying thickness of the bricks or the mortar bed, and with any false moves on the part of the bricklayer. It has been noticed that, in the long run, mortars of poor workability tend to give higher modulus of rupture in the wall test than more plastic mortars, doubtless because more pressure is required to push the bricks down to the line in those cases. On the other hand, plasticity seems to favor the bond in the couplet assembly where the same pressure is applied to all bricks. By and large, however, the tensile bond strengths from the couplets reflect the modulus values from the wall test, which in turn reflect, in some measure, the bond secured by the bricklayer in building a wall. For this reason the mechanized cross-brick couplet assembly, in its comparative freedom from effects of uncontrolled variables and its ready adaptation to different loadings, seems to offer the most satisfactory means of investigating bond strength of mortar to bricks.

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[5] R. W. Crum and H. W. Leavitt, "The Number of Specimens or Tests of Concrete and Concrete Aggregates Required for Reasonable Accuracy of the Average," Report on Significance of Tests of Concrete and Concrete Aggregates, Am. Soc. Testing Mats., p. 165 (1943). (Issued as separate publication.)

[6] These values were computed by the method given in the 1941 Manual on Presentation of Data, Am. Soc. Testing Mats., pp. 39-41. (Issued as separate publication.)

Acknowledgments:

This investigation, of which this paper is a highly condensed account, was very largely the work of H. G. Collins, R. F. Adams, J. E. Hentz and J. H. Laub. Special credit is due Mr. Collins for his design of the brick couplet assembly apparatus, and Mr. Laub for his many special tests of the mortars used. Mr. Adams, through his intimate knowledge of mechanics and his close scrutiny and careful record of all details, contributed largely to the success of the work, and without the aid of Mr. Hentz, a research-minded bricklayer of long experience, the investigation could hardly have been carried out at all.

DISCUSSION

Mr. J. W. McBurney.[1]—Mr. Pearson's paper is particularly timely and interesting in view of the protracted discussions in the Society's Committee C-12 on Mortar concerning the practicability of basing acceptance requirements for mortar upon tests of brick-mortar assemblages. His paper confirms the opinion that these types of tests require very careful control of technique to secure a reasonable degree of reproducibility.

Some years ago, at the National Bureau of Standards, determinations were being made of the tensile strength of cross-brick couplets as affected by difference in properties of bricks and of mortars. One operator, *M*, had been making and testing this type of specimen for about one year. An assistant, *N*, was assigned to work with him and, after one week's working with and training by operator *M*, the following experiment was tried. Twenty-eight bricks were picked at random from a shipment of Detroit commons. Using the same batch of mortar, each operator constructed couplets alternately, taking their pairs of bricks at random and continuing construction until both operators had completed 7 couplets. The experiment was repeated the next day using another random set of bricks from the same shipment and a batch of a mortar differing in composition from the batch previously used. The couplets were tested in tension after 28 days damp storage and the results are presented in the following tabulation:

Operator	Tensile Strength of Cross-Brick Couplets, psi.					
	Mortar A			Mortar B		
	Average	Max.	Min.	Average	Max.	Min.
M	45	64	22	43	57	31
N	27	52	0	18	42	9

The dispersion indicated by the maximum and minimum results for either operator is not surprising, since the bricks ranged in intitial rate of absorption from a minimum of 21 to a maximum of 64 g. (average 42 g.). The consistent difference between the two operators was unexpected, considering that operator *N* had been trained by and worked with operator *M*.

Mr. Pearson notes "an indefinite zone of little or no bond around the perimeter of the brick faces." As part of an investigation of bond between brick and mortar, permeability tests were made on panels $3\frac{3}{4}$ in. in thickness using a water solution of a dye. After completion of such tests, the brick-mortar joints were separated and the degree of penetration of the dye from the edges of the bricks was noted. Lack of contact between brick surface and mortar surface at their edges was observed on all combination of bricks and mortars which were examined. In some specimens these edge effects extended 1 in. into the joint. In few cases were they less than $\frac{3}{8}$ in. in depth. Tooling of joints appeared to have little effect. The effect of these openings upon flexural tests such as are illustrated in Fig. 1 of Mr. Pearson's paper will be obvious. Substituting a corrected depth determined by measurement after a dye penetration test for the gross depth might help the reproducibility of flexural tests on brick-mortar assemblages.

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[1] Senior Technologist, National Bureau of Standards, Washington, D. C.

Mr. Pearson's method of constructing cross-brick couplets is undoubtedly an improvement. It is questioned, however, whether edge effects will be completely eliminated even by that method. Pressure alone will not insure complete and even contact over the entire brick-mortar interface. Some years ago the experiment was tried of testing in compression a couplet consisting of two 15,000-psi. half bricks separated by a ½-in. joint of damp sand. The half bricks were very regular in shape and the sand joint was carefully leveled before placing the top brick. The upper and lower surfaces of the couplet were capped with plaster of Paris. One of the bricks started to crush at 3000 psi. The joint was reduced to ⅜ in. in thickness by this load and bearing was apparently limited to the area of a circle 2 in. in diameter. Within this circle the sand was squeezed dry. No appreciable pressure was exerted outside of this circle as judged by the lack of compaction of the sand. Excess sand flowed freely from between the bricks during application of the load.

Mr. Pearson is especially to be commended for his presentation of measures of the deviations of his results.

MR. F. O. ANDEREGG.[2]—Mr. Pearson's difficulties in getting satisfactory bond results are similar to those encountered in my laboratory. We went through the same experience, but by using water soluble dyes,[3,4] were able to overcome that effect, also noted by Mr. Pearson, "The mortar squeezes out more or less unevenly and leaves an indefinite zone of little or no bond around the perimeter of the brick faces. . . .".

It is respectfully suggested that Mr. Pearson has been testing notched specimens having variable and unknown notch dimensions. During the discussion of my paper last year, I warned against this danger.[5]

With perfect contact between brick and mortar the bond strength should be nearly as strong as the tensile strength of the mortar of the same water-cement ratio and degree of packing. At the apex of a notch, however, there occurs a concentration of stress, so that failure may develop at only a fraction of true bond value. This phenomenon may be illustrated by considering another brittle material, glass. Bulk glass has a tensile strength of about 10,000 psi., apparently due to the presence of numerous tiny flaws or notches. When care is taken to reduce their severity, as by drawing into fine fibers, strengths as high as 3,500,000 psi. have been obtained.[6] In view of this fact, increases in strength of 4 to 1, by eliminating notches, do not seem excessive, and our methods, by reducing scatter and uncertainty, are of value in comparing different cementing materials.

One conclusion which may be drawn from this discussion is that ordinary masonry construction is much weaker than it might be. In addition, it is not surprising that brick walls should leak as a rule, whenever they are struck by driving rains.

MR. J. C. PEARSON[7] (*author's closure, by letter*).—I wish to express my appreciation to Messrs. McBurney and Anderegg for emphasizing certain features in the assembly of brick and mortar bond specimens that are mainly responsible for erratic results in tests of such specimens. In general, I fully concur with them in their opinions, and particularly with Mr. McBurney in his remarks regarding the impracticability of basing acceptance tests for mortars on brick-mortar assemblages. On the other hand, Mr. Anderegg and I have been at odds for some time on the value of bond test results from the method he advocates, namely, the making of test couplets by first plastering both bricks with the mortar before pressing them together. Undoubtedly this method tends to eliminate the main cause of variations as occurring in the tests described in the paper, and from the standpoint of reproducibility alone is much better. But there remains the very pertinent question: Do the bond values obtained by this method bear any significant relation to the bonds in brick walls? In my own mind there are at least two reasons for doubt:

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[2] Consulting Specialist on Building Materials, New Haven, Conn.

[3] F. O. Anderegg, "Some Properties of Mortars in Masonry," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1130 (1940).

[4] F. O. Anderegg, "The Effect of Brick Absorption Characteristics upon Mortar Properties," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 821 (1942).

[5] Discussion of paper by F. O. Anderegg, *Ibid*, p. 835.

[6] F. O. Anderegg, "Strength of Glass Fibers," *Industrial and Engineering Chemistry*, Vol. 31, p. 290 (1939).

[7] Director of Research, Lehigh Portland Cement Co., Allentown, Pa.

1. The bond values so obtained are probably several hundred per cent higher than the average bond in brick structures.

2. If, according to Mr. Anderegg's implication, the bond strengths obtained by his method approach the tensile strengths of the mortars themselves, then the effect of plasticity on his results would be negligible or nearly so, whereas this property is regarded as of utmost importance in contributing to bond and watertightness in walls built with common bricks of medium or high absorption. It is still my belief that bond tests of bricks and mortar, to be of any practical value, ought to be patterned in such manner as to give results that bear some semblance to conditions in masonry structures. From this point of view there should not be too much objection to considerable deviations in test results, provided enough tests are made to indicate their reliability.

INCREASING THE REFLECTIVITY OF STANDARD PORTLAND CEMENT CONCRETES BY ADDITIONS OF HYDRATED LIME

By C. W. Muhlenbruch[1] and Ben Marcin[1]

Synopsis

It has been found that the reflectivity of standard portland-cement concretes, using river sand or white sand and river gravel, can be raised 15 percentage points by the addition of 30 per cent hydrated lime. Intermediate data indicate the relative reflectivities for various additions of lime. The decrease in reflectivity of specimens subjected to atmospheric exposure is slightly less than that found for white portland-cement concretes. After 25 cycles of freezing and thawing the white sand specimens, when mixed with any percentage of lime, show a slight loss in reflectivity. The effect of the addition of lime upon the compressive strength is also briefly discussed.

Continuing the work reported at the 1942 Annual Meeting of the Society,[2] additional studies have been made of the effect upon reflectivity of ordinary portland-cement concretes caused by the addition of hydrated lime. It was thought that the comparatively high cost of standard white portland cement may not be justified in some instances and that a reduced but yet sufficient reflectivity might be obtained with cheaper gray portland cement, hydrated lime, and a light colored fine aggregate.

The purpose of this investigation was to determine the influence of quantity of hydrated lime, type of fine aggregate, and various conditions of exposure on the reflectivity of standard portland-cement concretes.

Materials and Test Specimens

The portland cement used had the following general properties:

Normal consistency, per cent	27.0
Vicat setting time, hr.	
Initial	2.33
Final	4.50
Tensile strength, 7 days, psi	405
Compressive strength, 7 days, 2-in. cubes, psi	3300
Autoclave expansion, per cent	0.133
Specific surface, sq. cm. per g	1800

Allegheny River sand, which has a light brown color, and commercial white sand ground to a natural grain from a $99\frac{1}{2}$ per cent silica rock deposit near Mapleton, Pa., were the two fine aggregates studied. The following sieve analyses, made in conformity with A.S.T.M. Standard Method of Test for Sieve Analysis of Fine and Coarse

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[1] Assistant Professor of Civil Engineering, and Senior in Civil Engineering, respectively, Carnegie Institute of Technology, Pittsburgh, Pa.

[2] C. W. Muhlenbruch and E. H. Miller, "Factors Influencing the Reflectivity of White Portland Cement Concretes," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 775 (1942).

Aggregates (C 136 – 39)[3] were obtained for the fine aggregate:

	White Sand	River Sand
Percentage passing No. 4 sieve...	100	100
Percentage passing No. 8 sieve...	100	98
Percentage passing No. 16 sieve..	100	77
Percentage passing No. 30 sieve..	97	42
Percentage passing No. 50 sieve..	17	15
Percentage passing No. 100 sieve..	2	2

Since the previous investigation indicated that the coarse aggregate had no effect on reflectivity, Allegheny River gravel was substituted for the crushed limestone formerly used. This gravel had the following sieve analysis made in conformity with A.S.T.M. Method C 136 – 39.

Percentage passing 1½-in. sieve...........	100
Percentage passing 1-in. sieve............	96
Percentage passing ¾-in. sieve............	74
Percentage passing ½-in. sieve............	35
Percentage passing ⅜-in. sieve............	11
Percentage passing No. 4 sieve...........	3

In addition to the sieve analyses the following general properties were determined for all aggregates in accordance with the appropriate A.S.T.M. procedures:

	White Sand	River Sand	River Gravel
Unit weight, lb. per cu. ft.[a]...	94	101	108
Percentage voids[b]............	43	35	38
Apparent specific gravity[c].....	2.66	2.64	2.56
Fineness modulus[d]...........	1.84	2.66	2.81

[a] Standard Method of Test for Unit Weight of Aggregate (C 29 – 42), 1942 Book of A.S.T.M. Standards, Part II, p. 379.

[b] Standard Method of Test for Voids in Aggregate for Concrete (C 30 – 37), *Ibid.*, p. 381.

[c] Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 42), *Ibid.*, p. 371.

[d] Standard Specifications for Concrete Aggregates (C 33 – 42), *Ibid.*, p. 351.

The high-calcium hydrated lime used was added in the proportions of 10, 15, 20, or 30 per cent by weight of cement. This lime was *added* to the concrete mix and did not *replace* any cement.

Reflectivity specimens, 8 in. square and 2 in. thick, were cast in cardboard boxes which had been dipped in liquid Pyralin. When this Pyralin hardened on the cardboard, the surface presented to the setting concrete was less permeable than the paraffined cardboard previously employed and produced a better cast surface on the test specimen. A mix composed of one part by volume of standard portland cement, two parts by volume of fine aggregate, and three parts by volume of coarse aggregate with a water-cement ratio of 7 gal. per sack was selected as a base mix with which to compare subsequent limed mixes for the same degree of workability. When hydrated lime was added to the mixes in various proportions, it was necessary to increase the water content above that of the base mix to give a slump equal to the 4½-in. slump of the base mix. The quantity of fresh concrete required to cast the four test specimens prepared for the base mix constituted a batch. The weights of material required to yield one batch were then calculated by the absolute volume method to permit proportioning by weighing. Specimens were cured for 15 days at room temperature and humidity.

The tests for normal consistency, time of set, tensile strength, compressive strength of 2-in. cubes, autoclave expansion, and specific surface were made in conformity with A.S.T.M. Standard Methods of Sampling and Physical Testing of Portland Cement (C 77 – 40),[4] Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 – 37 T),[5] Tentative Method of Test for Autoclave Expansion of Portland Cement (C 151 – 40 T),[6] and Standard Method of Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 – 42).[7]

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[3] 1942 Book of A.S.T.M. Standards, Part II, p. 369.

[4] 1942 Book of A.S.T.M. Standards, Part II, p. 33.

[5] *Ibid.*, p. 1072.

[6] *Ibid.*, p. 1077.

[7] *Ibid.*, p. 47.

APPARATUS

The apparatus used to measure reflectivity was the same as that described previously.[2] Again it should be pointed out that reflectivity values differing by only a few per cent must be considered the same inasmuch as light measuring equipment will not permit comparisons better than about 3 per cent.

RESULTS AND DISCUSSION

Effect of Lime on Reflectivity of River Sand Concrete:

For a concrete made with standard gray cement, river sand, and river gravel the reflectivity of the cast surface was found to average 20 per cent and that of the troweled surface 25 per cent. When 30 per cent by weight of hydrated lime was added and the mixing water increased from 7 gal. per sack to approximately 9.1 gal. per sack, to produce the $4\frac{1}{2}$-in. slump of the base mix, the reflectivity of the cast surface was raised to 28 per cent and that of the troweled surface to 34 per cent. No further work was done with the river sand since these values of reflectivity were much less than those obtained with white sand.

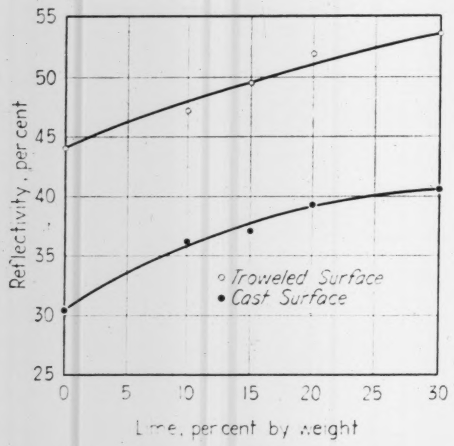

FIG. 1.—Effect of Additions of Hydrated Lime upon Reflectivity of Standard Portland Cement Concretes Made with White Sand. Each plotted point represents the average of four specimens.

Effect of Lime on Reflectivity of White Sand Concrete:

When the hydrated lime was added to the base concrete mix it was necessary to add water to produce a workability equivalent to that of the base mix. The slump test, made in accordance with A.S.T.M. Standard Method of Slump Test for Consistency of Portland-Cement Concrete (C 143 – 39),[8] was used to control the consistency. The average water-cement ratios, based on the actual and original quantity of cement used in the base mix and necessary to produce the proper consistency for each addition of lime, were as follows:

Quantity of Lime, per cent by weight	Water-Cement Ratio, gal. per sack
0	7.00
10	7.85
15	8.15
20	8.67
30	9.30

Figure 1 shows the effect of additions of hydrated lime upon the reflectivity of white sand, gray cement concretes. The 10 percentage points average increase in reflectivity for both troweled and cast surfaces for 30 per cent lime content is considered quite satisfactory. The maximum reflectivity of 53 per cent for the troweled surface of the 30 per cent lime specimen may be compared with an average of 61 per cent for white cement with river sand and 73 per cent for the white cement with white sand. Again it may be observed that the finishing operation with the trowel smooths out the surface and causes cement and lime particles to rise, producing a smooth and more reflective surface.

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[8] 1942 Book of A.S.T.M. Standards, Part II, p. 399.

Effect of Atmospheric Exposure on Reflectivity:

When the specimens were exposed to the atmosphere for 35 winter days, the average loss in reflectivity of troweled surfaces was 19 percentage points as may be seen from Fig. 2. When the blocks were surface cleaned with a brush and a mild soap solution, the average reflectivity regained was 2 percentage points.

This 40 per cent average loss, expressed as a percentage of the original reflectivity, for such exposures compares favorably with the 48 per cent loss observed for the same period of exposure for white cement, white sand specimens.[2] The small gain in reflectivity when the limed specimens were cleaned is comparable to that noted previously for a longer period of time for the white cement, white sand specimens.[9] The small gain does not compare very favorably, however, with the larger gain obtained for white cement, white sand specimens employing admixtures and exposed for a longer period of time.[9]

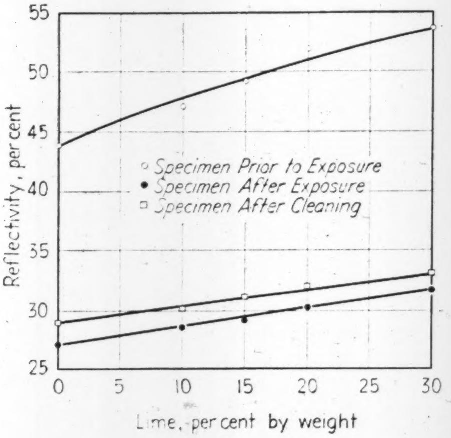

FIG. 2.—Effect of Atmospheric Exposure and Subsequent Cleaning upon Reflectivity of Standard Portland Cement Concrete Made with White Sand and Various Proportions of Lime. Each plotted point represents the average of two specimens.

Effect of Freezing and Thawing on Reflectivity:

Extreme temperature variations were again obtained by subjecting the blocks to a temperature of 20 F. for 12 hr. and then spraying them with tap water at 120 F. until they were surface warm. As may be seen from Fig. 3 this condition reduced the reflectivity of the troweled surface a uniform average of 4 percentage points and the cast surface a uniform average of 5 percentage points.

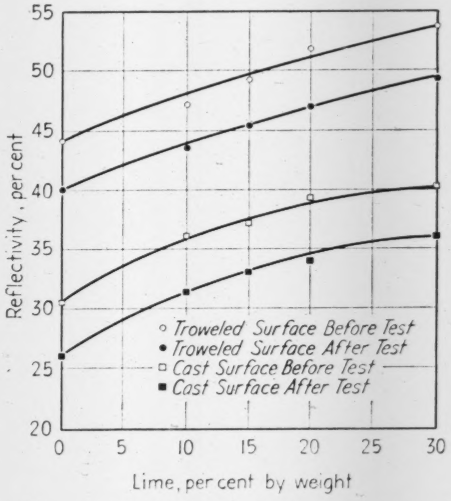

FIG. 3.—Effect of 25 cycles of Freezing and Thawing upon Reflectivity of Standard Portland Cement Concrete Made with White Sand and Various Proportions of Lime. Each plotted point represents the average of two specimens.

This average 8 per cent loss in reflectivity for the troweled surface, expressed as a percentage of the original reflectivity, compares very favorably with the 28 per cent loss observed for white cement, white sand specimens subjected to 35 similar cycles.[10] It appears that the concretes containing lime would generally be liable to less percentage of loss in reflectivity than any of the concretes made with white cement.

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[9] C. W. Muhlenbruch and E. H. Miller, "Factors Influencing the Reflectivity of White Portland Cement Concretes," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, Table I, p. 783.

[10] *Ibid.*, Table II, p. 784.

Effect of Additions of Lime on Compressive Strength:

Concrete compression test specimens were made and tested in conformity with A.S.T.M. Standard Method of Test for Compressive Strength of Concrete (C 39 – 42).[11] The test cylinders were moist cured for six days and tested on the seventh day.

As shown in Fig. 4, the 7-day compressive strength of the concrete was reduced by additions of hydrated lime. That this reduction in strength was caused by the increased water-cement ratio necessary to provide a workability equal to that of the base mix may also be seen from Fig. 4. By comparing the strength of similar concrete mixes having equal water-cement ratios but with or without lime, it may be seen that the decrease in strength follows the increase in water-cement ratio. Actually the higher percentages of lime show slightly higher strengths than the base mix. If high strength is required in addition to reflectivity it may be advisable not to use this method.

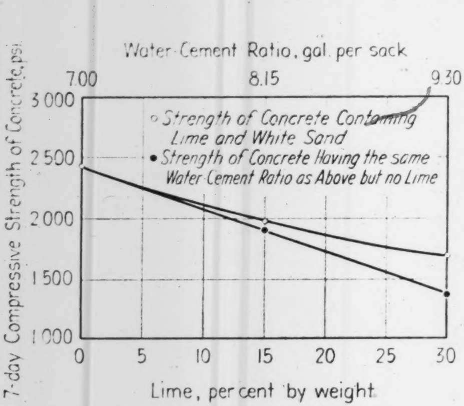

FIG. 4.—Effect of Additions of Hydrated Lime upon the 7-day Compressive Strength of Portland Cement Concretes with White Sand. Each plotted point represents the average of three specimens.

SUMMARY AND CONCLUSIONS

As a result of the test data presented in the paper the following conclusions seem justified:

1. Concrete of suitable, although not a maximum, reflectivity can be obtained using ordinary portland cement, a light colored fine aggregate, and hydrated lime.

2. When 30 per cent by weight of hydrated lime is added to ordinary gray cement concrete made with river sand, the reflectivity of the troweled surface may be expected to increase from a reflectivity of 25 per cent to a reflectivity of 34 per cent. When the same quantity of lime is added to an ordinary concrete made with white sand the reflectivity of the troweled surface may be increased from a reflectivity of 44 per cent to a reflectivity of 53 per cent.

3. When subjected to atmospheric exposure, ordinary concrete containing lime may decrease 40 per cent in reflectivity but not as much as concrete made with white cement.

4. Severe cycles of freezing and thawing will reduce the reflectivity of cement concrete containing lime. The average reduction of 8 per cent is considerably less than that observed for concretes made with white cement.

5. To secure workability, it is necessary to increase the amount of mixing water in concretes containing lime. The decrease in compressive strength with this increase in water-cement ration is slightly less than that of mixes having the same water-cement ratio but no lime.

Acknowledgment:

The authors gratefully acknowledge the help and cooperation given this investigation by the National Lime Association, Washington, D. C., who sponsored the work as an "Industrial Research Thesis" in the Materials Testing Laboratory of the Carnegie Institute of Technology.

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[11] 1942 Book of A.S.T.M. Standards, Part II, p. 391.

STUDIES TO DEVELOP AN ACCELERATED TEST PROCEDURE FOR THE DETECTION OF ADVERSELY REACTIVE CEMENT-AGGREGATE COMBINATIONS

BY THOMAS E. STANTON[1]

SYNOPSIS

Considerable research has been carried on during recent years with relation to the phenomenon of the excessive expansion of concrete apparently traceable to some chemical reaction between the cement and aggregate.

In California one cause has been definitely traced to a reaction between the alkalies in the cement (Na_2O and K_2O) and an opaline constituent of the aggregate. The underlying cause of the expansive reaction in other cases is obscure, at least in so far as positive identification of the reactive mineral in the aggregate is concerned.

In any case, and whatever the cause, it is very desirable that some accelerated test procedure be developed to determine the potential expansion characteristics of any cement-aggregate combination.

Tests discussed in this report include (1) chemical analyses of both aggregate and cement, (2) mineralogical and petrographic analyses, (3) fluorescence under ultraviolet light, (4) reaction of cement-aggregate combinations in caustic solutions, (5) dynamic (sonic) modulus changes with relation to expansion, (6) cement content, and (7) curing procedure.

Best over-all long-time results were obtained through curing in sealed containers at 70 F. For accelerated short-time tests curing at a temperature of 110 F. either in a sealed container or in a sodium hydroxide solution is indicative of long-time results at 70 F.

It should be clearly understood that these tests and the resultant conclusions are primarily based on tests with California reactive opal bearing aggregates.

CHEMICAL ANALYSES

Cement:

In any reactive combination of the type discussed in this report the short-time expansion is, in general, a function of the total alkali content of the cement ($Na_2O + K_2O$) expressed as Na_2O. Low-alkali cements ($\frac{1}{2}$ per cent or less) almost invariably develop little early expansion at ordinary room temperatures (70 F. ±), whereas excessive early expansion (0.1 per cent or greater) is almost invariably traceable to the use of a cement containing in excess of 0.5 per cent total alkali.

A very simple solution would, therefore, appear to be a limitation on the alkali content of the cement, thus obviating the necessity for any more extensive and expensive tests.

The problem is not so simple, however, for the reason that although the alkali in cement may always act to some extent on any aggregate, this reaction in the case of most commercial aggregates may not result in the formation of reaction products of a disruptive nature.

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Therefore, the mere fact that a cement has a high alkali content cannot in the light of present evidence be considered as conclusive evidence of probable early or even ultimate adverse reaction, regardless of the aggregate type, and some other test such as one of cement and aggregate in combination is necessary. About all that can be said at the present time is that in any particular case no other tests than a chemical analysis of the cement appear necessary in the case of a definitely low-alkali cement but with high-alkali cements further tests are essential, if there is any reason to suspect the aggregate.

Aggregate:

Chemical analyses of the aggregate have not been very informative.

As previously stated, the source of most of the trouble in California is opal which need be present to the extent of only 1 per cent or less of the aggregate to be the cause of considerable trouble when in combination with a high-alkali cement. It is impracticable to detect such small percentages of opal by chemical analysis and, therefore, this method appears unavailable as an accelerated test procedure.

Sodium Hydroxide Solubility of Aggregate:

Considerable study has been made of the reaction characteristics of aggregates with relation to solubility in sodium hydroxide, but so far no sufficiently sensitive procedure has been developed which has practical application to the problem in hand.

Unreactive minerals such as quartz, chalcedony, feldspar, amphibules, mica, and many of the other common rock-forming minerals are attacked over a period of time by a 10-per cent sodium hydroxide solution.

It is possible that a weaker solution applied to an aliquot part of the entire sample may produce results which mean something. This is a field for further study, although the sodium hydroxide solubility method of analysis does not appear to offer much encouragement.

MINERALOGICAL AND PETROGRAPHIC ANALYSES

A petrographic study has been made of several types of reactive aggregates in an endeavor to isolate the reactive from the nonreactive. The results thus far indicate that petrographic analyses are both positive and reliable for the detection of opaline constituents in aggregates.

However, this method for detection of presumably reactive andesite, rhyolite, phyllite, or other types of aggregate not containing opal, has not, up to the present time, been found very satisfactory. A thorough and complete petrographic study procedure may some day be developed for detecting reactive andesites and other reactive aggregates of a non-opaline type. Our researches thus far with the problem of andesite lead us to believe the glassy groundmass in certain types, in a partial state of alteration or devitrification, is the reactive phase.

FLUORESCENCE UNDER ULTRAVIOLET LIGHT

Continued experience with the use of ultraviolet light as a test for detection of reactive aggregates (combined or uncombined in concrete) has produced no change in the original conclusion that this test is entirely too erratic and inconsistent to be reliable.[2] Examinations have been made of concrete cores, cylinders, expansion bars, and aggregates under both a low and a high wave length ultraviolet light. Our findings may be briefly summarized as follows: (*a*) many nonreactive aggregates fluoresce both before and after incorporation in concrete; (*b*) fluorescent zones around aggregates in concrete are not always zones of gel formation; (*c*) gel areas in some concretes are strongly fluorescent, whereas in other concrete samples no fluorescence of the gel is observed; and (*d*) different cements produce different fluorescent properties with the same type of fluorescent reactive aggregates.

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[2] *Proceedings*, Am. Soc. Civil Engrs., September, 1941, p. 119; Table 14, p. 114.

REACTION OF CEMENT-AGGREGATE COMBINATIONS IN CAUSTIC SOLUTIONS

A rapid method for the detection of reactive aggregate by gel formation was discussed by the author in the *Journal* of the American Concrete Institute (Supplement), November, 1942, page 236-31, *et seq.*

As originally developed, the method was used principally for detection of reactivity of ledge rock or large size gravel aggregate samples, crushed to about ½ to ¾-in. size. Similar tests were later made with fine aggregates and pea gravels. Results to date have been quite satisfactory for the rapid detection of the opaline content in any aggregate.

The method consists of the following steps:

1. The sample, if ledge rock, or a large piece of gravel aggregate, is crushed to minus ⅜ in. to plus No. 4 sieve size.

2. If the material is a fine aggregate, the entire graded sample is used, although it may occasionally be desired to scalp the aggregate on some particular size in order to determine the greatest concentration of opaline constituents above or below a certain grading size.

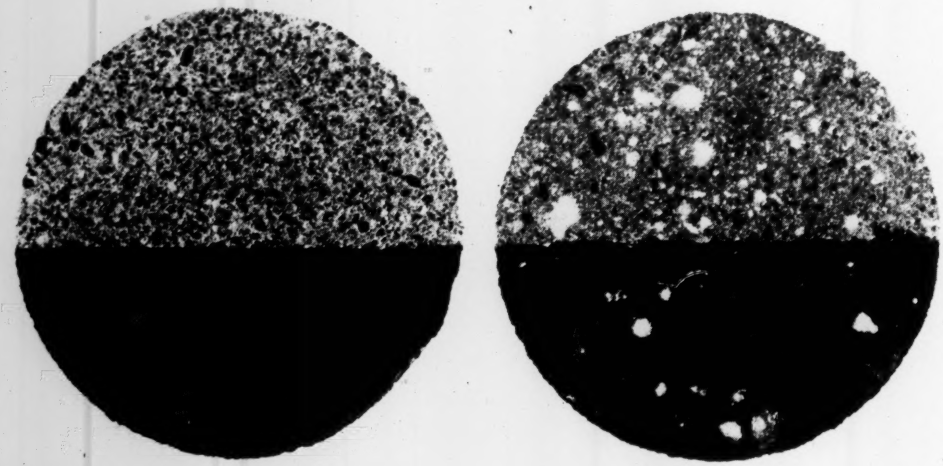
3. The sample is then treated as follows: If a fine aggregate, it is mixed with a fairly high-alkali cement in a 1:2 mix by dry rodded volume, and cast into pats of ¾ to 1-in. thickness.

In the case of larger size particles of crushed aggregates, the pieces are embedded about ⅛ in., or until submerged, in a neat cement slurry and allowed to set.

4. The pats are cured in a fog room for 48 hr., then removed from the molds and the top surface lapped at moderate speed with No. 220 carborundum, sufficiently to remove the surface layer of neat cement and expose the grains.

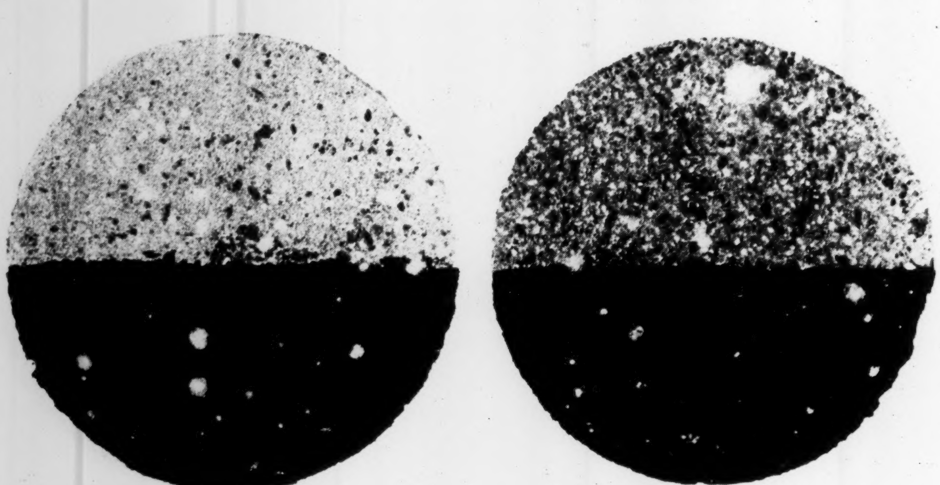
5. The pats are then stored in a lead-lined tank for an indefinite period in a caustic solution composed of one part by volume of 0.5 *N* sodium hydroxide, one part potassium hydroxide, and two parts saturated limewater ($Ca(OH)_2$). Addition of the sodium and potassium hydroxides causes precipitation of the lime; therefore, these salts are added to clear saturated limewater and the mixture allowed to stand for 3 hr., after which the solution is filtered until clear. The tops of the pats during immersion are covered by at least ¾ to 1 in. of solution. A glass cover is wax-sealed over the top of each tank to prevent excessive evaporation and carbonation of the solution.

To facilitate observation of the gel areas, the surface of all or a portion of the pat is lightly coated with carbon black in denatured alcohol. This affords a more distinctive background for the white gel which passes through with ease and without becoming discolored. Daily observation of the pats is made during the immersion period, which, of course, varies with the type of aggregate. For the California opaline aggregates usually 3 to 5-days immersion at room temperature is sufficient to produce a white gelatinous exudation in or around the reactive opaline particles, Fig. 1. Some samples having a small amount of less-reactive opal may require 1 or 2 weeks, at ordinary room temperature.



(*a*) Unreactive Coyote Sand similar to that used in the pavement north of Bradley, Monterey County.

(*b*) Reactive Oro Fino Sand similar to that which caused extreme expansion of the pavement north of Bradley.



(*c*) Sand from Los Angeles County, Lomita District.

(*d*) Saticoy Sand, Ventura County.

Fig. 1.—Showing Reaction in a Caustic Solution of a Neutral Sand and Three Reactive Commercial Sands Containing Opal.

A modification of the above test involves keeping one series of pats immersed at a temperature of about 130 F. over a period of time, while a duplicate series is immersed in another container at 70 F. Results indicate that the high temperature enormously accelerates the formation of the gelatinous exudation. Approximately the same quantity of gel is found within 16 or 17 hr. at 130 F. as in 1 week at 70 F. The test, however, has thus far proven unsatisfactory for nonopaline aggregates and is, as far as known, a specific test for opal only. Although it does not tell how potentially bad an aggregate containing opal may be, it does afford a quick means of ascertaining whether an aggregate is of the opaline type, and herein lies its greatest value.

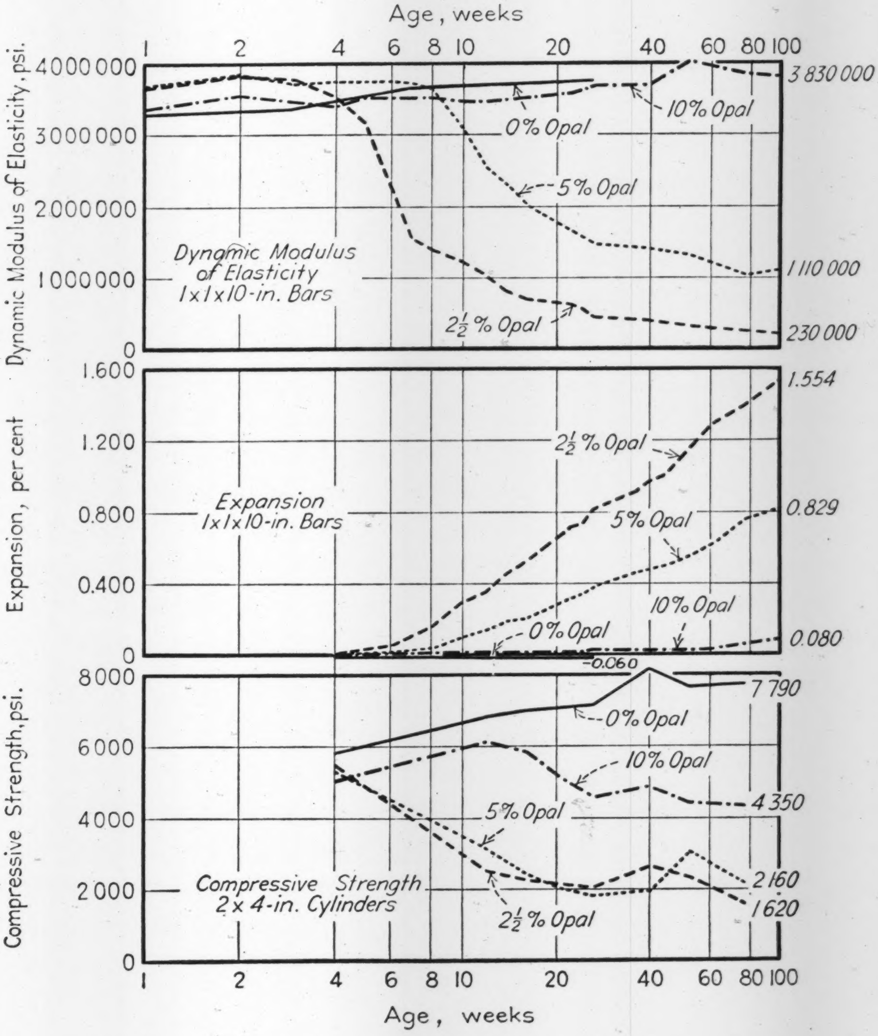


FIG. 2.—Dynamic Modulus of Elasticity, Expansion, and Compressive Strength of 1:2 Mortar Specimens.

High alkali (1.14 per cent) cement, "GS" + Russian River sand + 0 to 10 per cent of thirty to eighty mesh opal from dredger dumps near Roseville, Calif. All specimens cured in sealed containers.

This "pat" test has been employed on many occasions to decide whether to make an extensive series of expansion tests on an unknown aggregate, or whether to make only a few. It has been found to be a quicker and more reliable way of detecting the presence of small amounts of opal in aggregate samples than by petrographic methods. Several dozen tests have been made in the past few months on various commercial sands from within California, and several sands from other states. Several materials have revealed a gelatinous exudation, later proven to be opal, where no opal was originally observed by petrographic analysis. In these cases the actual amount of opal by weight in the entire sample would, if possible to determine, be of an extremely small order, probably less than 0.1 per cent.

RELATION OF DYNAMIC MODULUS TO EXPANSION

The possibility of detecting probable adverse action more readily and at earlier stages by means of dynamic modulus of elasticity determinations has been thoroughly explored.

Figure 2 illustrates the results of the earliest studies along this line. Numerous later tests are in entire agreement with the results shown in this figure.

It will be noted that while the dynamic modulus as well as the expansion and compressive results after 12 weeks are consistent with relation to percentage of reactive mineral, the modulus at 8 weeks in the case of the highly reactive combination with 5 per cent opal is substantially the same as for the control specimens which contained no opal, whereas there had been marked expansion of the same 5 per cent opal specimens in the same period.

It will be noted that with 10 per cent opal there was no drop in dynamic modulus up to 100 weeks even though there was some increase in expansion and a definite drop in compressive strength.

Similar phenomena have been noted in numerous later tests where even with a substantial expansion the dynamic modulus although at first falling off with increasing expansion ultimately showed at least a partial recovery.

It is, therefore, concluded that no dependence can be placed on dynamic modulus measurements at early ages and possibly even at later ages for combinations that are objectionably though not so highly reactive as those shown in Fig. 2.

It might be assumed, as has been suggested by some, that with moderate expansion any increase in dynamic modulus with age is indicative of autogenous healing and that in such cases there is little permanent injury to the concrete.

To test this theory and, at the same time, to test another theory—that increase in dynamic modulus, in the case of some reactive combinations, may be due to the formation of a silica gel which serves to maintain the resonant or vibration frequency—mortar bar specimens of a combination of a highly reactive aggregate and a low-alkali cement which had shown shrinkage instead of expansion at 3 yr. were tested for dynamic modulus and for modulus of rupture. The broken ends were then cemented together with sodium silicate and retested for dynamic modulus at different ages with the following results:

Age	Shrinkage, per cent	Modulus of Rupture, psi.	Dynamic Modulus, psi. × 10^{-6}	
			Specimen No. 1	Specimen No. 2
TEST AT 3 YR. ON A REACTIVE-AGGREGATE - LOW-ALKALI CEMENT COMBINATION				
	0.0120	1404	4.69	4.69
TESTS FOR DYNAMIC MODULUS AFTER CEMENTING BROKEN ENDS TOGETHER WITH SODIUM SILICATE				
24 hr.	...	..	2.66	3.17
10 days	...	...	4.07	4.19
31 days	...	...	4.37	4.44

While the apparent modulus as determined from the vibration frequency was high at 31 days after cementing with sodium silicate, the bending strength was nil, specimens subsequently breaking under their own weight.

There is, therefore, good reason for discarding the dynamic modulus method for determining potentially reactive characteristics at early ages at least, and considerable doubt as to the reliability of this method as an alternate method for detecting moderately reactive combinations at any age.

CEMENT CONTENT

All tests show that the expansion at early ages is a function of the cement content and that in any adversely reactive combination there is a markedly greater expansion of a 1:2 mortar than a 1:3, and usually a greater expansion of a 1:1 than a 1:2. However, with some highly reactive combinations the ultimate expansion appears greater with the 1:2 than the 1:1 mix.

This is another example of the phenomenon that excesses sometimes have a reverse effect. Thus an excess of reactive particles in the aggregate may result in little, if any, expansion. The same condition may develop when the alkali in the cement is present in what may be an excess with relation to the reactivity of the aggregate.

It has been noted that a relatively low-alkali normally unreactive cement may become quite active in mortars of equal proportions of cement and a reactive fine aggregate.

It would, therefore, appear undesirable to conduct routine tests at higher cement contents than 1:2.

CURING PROCEDURE

Sealed Containers:

Since earlier work showed the need of storing the 1 by 1 by 10-in. specimens in sealed containers all are now placed in closed metal containers with a small amount of water in the bottom of the container to maintain a humid atmosphere. The cover is usually sealed with adhesive tape.

As conducted up to the present time the specimens which are stood on end in the container are reversed at each measuring period and the water in the bottom of the container is changed at least as frequently. The measuring plugs in each end of the specimen project approximately $\frac{1}{2}$ in. beyond the ends of the specimens and as there is never more than approximately $\frac{1}{4}$ in. of water in the bottom of the container the ends of the specimens are never immersed in the water. Any closed type of container can be used and it is, therefore, not necessary to go into any further details.

Even with the precautions taken to keep all parts of the specimen out of the water it has been noted that moisture accumulates on the sides of the specimens and runs to the lower end. This has given rise to the theory in some quarters that there may be a leaching of salts from the upper end of a specimen and a corresponding deposit on the lower end which may increase the alkali content to such an extent, in the case of a low-alkali cement, as ultimately to cause expansion, thereby accounting for the fact that high expansion is sometimes observed after long exposure even in the case of low-alkali cement - reactive aggregate combinations. This phase of the subject has never been investigated in the California studies. Generally consistent results have been secured when specimens are cured in the manner described, definitely reactive combinations always developing substantial expansion in comparatively short periods, even at normal temperatures, and nonreactive combinations consistently showing little, if any, expansion over a period of years.

The necessity for storing in sealed containers holds good even for 2 by 2-in. cross-section specimens, but larger concrete specimens, either 6 by 12-in. cylinders or 6 by 6-in. beams, may develop greatest expansion when immersed in water.

Curing Temperature:

Early tests were all made in sealed containers at normal laboratory temperatures averaging 70 F.; highly reactive cement-aggregate combinations reacting readily and in a comparatively short time at this temperature. Less but still ultimately excessively reactive combinations require a much longer period.

Since Carlson[3] first directed attention to temperature as a factor,[4] extensive temperature studies have been made by a number of agencies, particularly by the Bureau of Reclamation, the Public Roads Administration, and Bailey Tremper of the Washington State Highway Department.

Our own experience covering hundreds of determinations indicates that while there may be an optimum temperature this temperature may be different for each reactive cement-aggregate combination and that the total ultimate expansion appears to be a function of both temperature and reactive characteristics.

A complication is encountered with tests at high temperatures in that some low-alkali cements normally unreactive at 70 F. and possibly even at 110 F. become highly reactive at 130 F. We must be careful, therefore, that in the search for an accelerated test procedure we do not produce a reaction which never develops under job conditions.

While it is true that in many localities the surface of a concrete mass may be heated by the sun to as high as 130 F., the interior of the mass, after the original heat of hydration subsides, seldom reaches temperatures much, if any, in excess of 100 F. Furthermore temperatures even as high as 100 F., with attendant appropriate moisture conditions inducing the formation of reaction products may exist during only a fractional part of each year.

TABLE I.—EXPANSION OF COMMERCIAL SANDS FROM ALL PARTS OF CALIFORNIA IN COMBINATION WITH A HIGH-ALKALI CEMENT. AGE OF SPECIMENS, TWELVE MONTHS.

1 by 1 by 10-in. bars cured in sealed containers at temperatures indicated.

Sand	High Alkali Cement (1.07% Na_2O). Expansion, per cent		Sand	High Alkali Cement (1.07% Na_2O). Expansion, per cent	
	70 F.	130 F.		70 F.	130 F.
No. 28060	0.034	0.147	No. 28090	0.019	0.049
No. 28061	0.032	0.063	No. 28091	0.011	0.017
No. 28062	0.029	0.028	No. 28092	0.022	0.016
No. 28063	0.027	0.142	No. 28093	0.022	−0.003
No. 28064	0.029	0.052	No. 28094	0.020	0.005
No. 28067	0.026	0.050	No. 28095	0.035	0.001
No. 28068	0.020	0.100	No. 28096	0.005	0.019
No. 28069	0.016	0.041	No. 28097	0.035	0.032
No. 28070	0.026	0.191	No. 28098	0.013	0.062
No. 28071	0.031	0.058	No. 28099	0.016	0.027
No. 28072	0.022	0.072	No. 28100	0.022	0.015
No. 28073	0.024	0.033	No. 28101	0.007	0.008
No. 28074	0.346	0.493	No. 28102	0.046	0.173
No. 28074-A	0.338	0.498	No. 28103	0.013	0.042
No. 28075	0.014	0.128	No. 28104[a]	1.193	1.547
No. 28076	0.015	0.131	No. 28104-A[a]	1.438	1.209
No. 28077	0.011	0.009	No. 28105[b]	0.898	0.282
No. 28078	0.012	0.084	No. 28105-A[b]	0.998	0.635
No. 28079	0.022	0.025	No. 28107-A[c]	1.556	0.978
No. 28080	0.035	0.463	No. 28107-B[c]	1.046	1.397
No. 28081	0.011	0.102	No. 28107-C[c]	0.050	−0.004
No. 28082	0.006	0.086	No. 28109	0.009	0.091
No. 28083	0.012	0.061	No. 28110	0.246	0.354
No. 28084	0.010	0.011	No. 28111	0.026	0.076
No. 28085	0.014	0.033	No. 28112	0.017	0.123
No. 28086	0.016	0.019	No. 28113	0.024	0.063
No. 28087	0.008	0.026	No. 28116	0.154	0.126
No. 28088	0.012	0.024	No. 28118	0.012	0.030
No. 28089	0.016	0.022	No. 28119	0.015	0.153

[a] Sand No. 28104 composed of a mixture of No. 28082 and 2½ per cent opaline chert No. 28038.

[b] Sand No. 28105 composed of a mixture of No. 28082 + 10 per cent opaline chert No. 28039.

[c] Sand No. 28107-A composed of sand No. 28082 + 2½ per cent No. 28038.

Sand No. 28107-B composed of sand No. 28082 + 5 per cent No. 28038.

Sand No. 28107-C composed of sand No. 28082 + 10 per cent No. 28038.

Therefore, while temperature as an accelerator may be an excellent tool, it should not be relied upon to the exclusion of all other factors; and if high expansion develops at either 110 or 130 F. such development should be considered as a warning rather than as conclusive evidence without further study.

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[3] R. W. Carlson, Associate Professor of Civil Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[4] *Proceedings*, Am. Soc. Civil Engrs., February, 1941.

In Table I are shown the 12-month expansion data for a large number of commercial sands from all areas of California. This series was made for the dual purpose of securing data relative to the reactive characteristics of any sands which might be used in state work and further data on expansion at 130 F. to compare with that at 70 F.

Sets Nos. 28104, 28105, and 28107, as will be noted from the footnotes, are synthetic specimens composed of an unreactive sand to which was added small percentages of two types of known reactive minerals. The rest of the specimens can be divided into two groups: (*a*) Those which show little, if any expansion in 12 months at 70 F., and (*b*) those which expand more than 0.1 per cent in the same period of time.

In Table II are shown some short-time tests as well as tests at 12 months, on the most reactive combinations listed in Table I.

TABLE II.—COMPARATIVE EXPANSIONS OF SOME HIGH-ALKALI (1.07% Na_2O) CEMENT - REACTIVE AGGREGATE COMBINATIONS AT 70 F. AND 130 F.

1 by 1 by 10-in. bars—1:2 mortars—cured in sealed containers.

Aggregate	Identification	Temperature, deg. Fahr.	Expansion, per cent						
			28 Days	2 Months	3 Months	4 Months	5 Months	6 Months	12 Months
No. 28074.........	Saticoy	70 130	0.013 0.146	0.022 0.229	0.024 0.352	0.087 0.449	0.145 0.462	0.210 0.464	0.346 0.493
No. 28074A	Saticoy (Repeat)	70 130	0.020 0.199	0.028 0.296	0.051 0.334	0.095 0.376	0.138 0.402	0.178 0.440	0.338 0.498
No. 28080.........	Truckee River	70 130	0.002 −0.014	0.009 −0.015	0.010 0.030	0.003 0.091	0.014 0.167	0.016 0.218	0.035 0.463
No. 28102.........	San Joaquin River	70 130	0.007 0.066	0.012 0.102	0.011 0.097	0.015 0.103	0.015 0.115	0.019 0.117	0.046 0.173
No. 28104.........	28082 + 2½% Opaline Chert No. 28038	70 130	−0.006 0.101	0.006 0.947	0.090 1.454	0.330 1.484	0.429 1.494	0.634 1.519	1.193 1.547
No. 28104A.........	28082 + 2½% Opaline Chert No. 28038	70 130	0.002 0.081	0.021 0.429	0.092 0.711	0.519 0.988	0.671 1.049	0.861 1.111	1.438 1.209
No. 28105.........	28082 + 10% Opaline Chert No. 28039	70 130	0.018 0.237	0.397 0.262	0.519 0.265	0.596 0.269	0.664 0.269	0.775 0.270	0.898 0.282
No. 28105A	28082 + 10% Opaline Chert No. 28039	70 130	0.043 0.352	0.434 0.476	0.549 0.506	0.688 0.546	0.773 0.566	0.847 0.572	0.998 0.635
No. 28107A	28082 + 2½% Opaline Chert No. 28038	70 130	−0.002 0.043	0.078 0.368	0.352 0.514	0.655 0.710	0.899 0.779	1.156 0.916	1.556 0.978
No. 28107B	28082 + 5% Opaline Chert No. 28038	70 130	−0.001 −0.018	0.053 0.027	0.181 0.030	0.328 0.071	0.383 0.085	0.500 0.366	1.046 1.397
No. 28107C.........	28082 + 10% Opaline Chert No. 28038	70 130	0.008 −0.009	0.017 0.002	0.032 −0.022	0.034 −0.019	0.046 −0.017	0.047 −0.008	0.050 −0.004
No. 28110.........	Sidebotham L. A. Co. Pea Gr. Crushed to Sand Size	70 130	0.002 0.224	0.008 0.268	0.040 0.316	0.088 0.328	0.137 0.338	0.166 0.337	0.246 0.354
No. 28116.........	Cowlitz, Oregon	70 130	0.002 0.017	0.007 0.048	0.010 0.066	0.025 0.079	0.050 0.086	0.077 0.085	0.154 0.126
No. 28136.........	Oro Fino	70 130	... 0.021	... 0.060	... 0.065	... 0.094	... 0.095	... 0.098	... 0.120
No. 28137.........	Piru	70 130	... 0.190	... 0.316	... 0.380	... 0.456	... 0.538	... 0.581	... 0.676
No. 28138.........	Sisquoc	70 130	... 0.011	... 0.022	... 0.049	... 0.086	... 0.128	... 0.162	... 0.182

Attention is called to the fact that with some of these more reactive combinations the ultimate expansion at 70 F. is greater than at 130 F. As a rule, however, temperature is definitely an accelerator at early ages and, therefore, the fact that ultimate expansion at 70 F. may be greater than at 130 F. does not alter the value of temperature for accelerating any probable reaction.

In addition to the more reactive combinations the sands in Table I (Fair Oaks, Stony Creek, Grant Pacific Lindsay, South Fork Trinity River, Hoopa, Truckee River, Reno, Kaiser Radum, San Joaquin River, Herndon) show comparatively low expansions in 12 months at 70 F. but definitely high at 130 F. Of these only two come from sources that are suspected; namely, No. 28080 from the Truckee River at Reno, Nev., and No. 28102 from the San Joaquin River near Herndon, Fresno County. Sand from another San Joaquin River source was used with a high-alkali cement in a traffic circle at Fresno in which considerable expansion and map cracking has developed.[5] Prior to this test series the Truckee River sand had not been suspected, but recent inspection has been made of structures in which this sand and a high-alkali cement was used. Typical crazing and map cracking indicative of excessive expansion was found.

It will be noted (Table II) that excessive expansion at 130 F. did not become apparent until after three months in the case of the Truckee River sand, whereas excessive expansion developed in 28 days with the San Joaquin River sand. There is considerable evidence that an andesitic rock may be at the base of the trouble in these two sands, whereas the reactive mineral in the highly reactive California aggregates is undoubtedly opal.

TABLE III.—COMPARISON OF EXPANSION CHARACTERISTICS OF 1:2 MORTARS COMPOSED OF A HIGHLY REACTIVE FINE AGGREGATE AND TWENTY-SEVEN CEMENTS FROM DIFFERENT SECTIONS OF THE UNITED STATES.

Reactive aggregate consisted of a blend of a neutral sand and 10 per cent of the reactive siliceous magnesian limestone No. 28039, which contains approximately 20 per cent opal.

These tests were run not only to determine the reaction characteristics of the cements but likewise to determine the effect of temperature on the rate of reaction.

Cements are arranged in order of increasing total alkali as Na_2O.

Cement	Per Cent Alkali as Na_2O	Temperature, deg. Fahr.	Expansion, per cent						
			28 Days	2 Months	3 Months	4 Months	5 Months	6 Months	12 Months
No. 1	0.17	70	−0.002	0.001	0.001	0.001	0.000	0.000	−0.004
		110	−0.003	0.001	−0.002	−0.002	−0.001	−0.002	0.018
		130	−0.001	0.002	0.002	0.002	0.008	0.008	0.327
No. 2	0.21	70	−0.008	−0.005	−0.005	−0.004	−0.003	−0.005	−0.007
		110	−0.016	−0.013	−0.013	−0.005	−0.006	−0.011	−0.010
		130	−0.005	0.002	0.000	0.004	0.008	0.011	0.224
No. 3	0.22	70	0.002	0.002	−0.002	0.001	0.003	0.002	0.012
		110	0.000	0.007	0.022	0.034	0.059	0.116	0.387
		130	0.002	0.002	−0.002	0.000	0.005	0.002	0.273
No. 4	0.23	70	0.004	0.005	0.002	0.004	0.007	0.006	0.006
		110	−0.006	−0.006	−0.010	−0.005	−0.005	−0.004	−0.003
		130	0.001	0.007	0.002	0.005	0.008	0.012	0.078
No. 5	0.23	70	−0.012	−0.014	−0.015	−0.012	−0.013	−0.015	−0.018
		110	−0.021	−0.018	−0.014	−0.012	−0.012	−0.015	−0.012
		130	−0.016	−0.003	0.055	0.145	0.200	0.241	0.498
No. 6	0.33	70	0.004	0.004	0.006	0.005	0.006	0.006	0.009
		110	0.009	0.023	0.093	0.193	0.318	0.450	0.761
		130	0.003	0.006	0.035	0.099	0.131	0.330	0.634
No. 7	0.34	70	0.004	0.004	0.004	0.006	0.015	0.014	0.026
		110	0.006	0.007	0.022	0.032	0.051	0.068	0.309
		130	0.004	0.010	0.009	0.024	0.044	0.060	0.352

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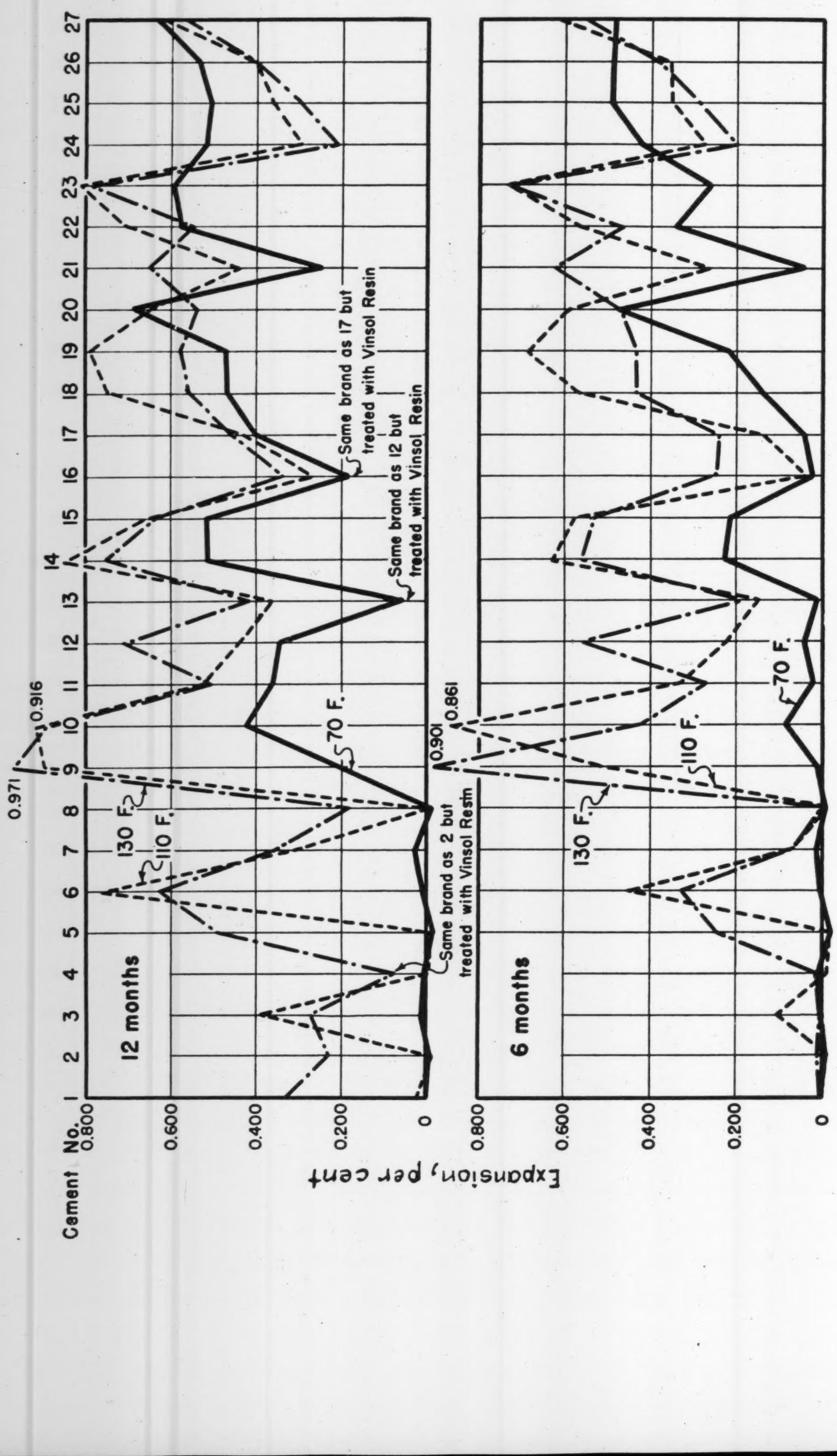
[5] *Journal*, Am. Concrete Inst., January, 1942, p. 211.

TABLE III—*Continued.*

Cement	Per Cent Alkali as Na_2O	Temperature, deg. Fahr.	Expansion, per cent						
			28 Days	2 Months	3 Months	4 Months	5 Months	6 Months	12 Months
No. 8	0.36	70	−0.007	−0.004	−0.003	−0.004	−0.003	−0.004	−0.007
		110	−0.016	−0.012	−0.013	−0.004	−0.008	−0.010	−0.010
		130	−0.008	−0.001	−0.003	−0.002	0.001	0.002	0.185
No. 9	0.37	70	−0.009	−0.006	0.005	0.004	0.006	0.010	0.213
		110	−0.006	0.019	0.098	0.279	0.367	0.519	0.894
		130	0.004	0.200	0.732	0.844	0.878	0.901	0.971
No. 10	0.46	70	0.003	0.088	0.019	0.030	0.055	0.088	0.420
		110	0.008	0.107	0.304	0.620	0.814	0.861	0.916
		130	0.006	0.049	0.175	0.253	0.330	0.420	0.696
No. 11	0.48	70	0.001	0.003	0.004	0.007	0.010	0.022	0.359
		110	0.003	0.010	0.088	0.224	0.260	0.339	0.518
		130	0.013	0.030	0.064	0.127	0.211	0.272	0.502
No. 12	0.48	70	0.001	0.007	0.007	0.015	0.024	0.042	0.344
		110	0.006	0.014	0.070	0.137	0.188	0.222	0.434
		130	0.010	0.051	0.316	0.311	0.463	0.566	0.709
No. 13	0.49	70	−0.002	0.000	0.001	0.003	0.009	0.011	0.065
		110	0.000	0.006	0.042	0.103	0.128	0.149	0.359
		130	0.003	0.017	0.052	0.121	0.158	0.188	0.414
No. 14	0.50	70	0.000	0.024	0.081	0.165	0.192	0.230	0.512
		110	0.008	0.127	0.328	0.543	0.592	0.626	0.841
		130	0.001	0.038	0.216	0.359	0.461	0.566	0.752
No. 15	0.51	70	−0.004	0.003	0.069	0.160	0.185	0.218	0.515
		110	0.005	0.076	0.231	0.420	0.520	0.579	0.656
		130	0.017	0.135	0.272	0.336	0.413	0.537	0.633
No. 16	0.52	70	−0.001	0.007	0.009	0.011	0.019	0.026	0.183
		110	−0.002	0.000	0.006	0.014	0.029	0.034	0.261
		130	0.004	0.027	0.079	0.182	0.219	0.244	0.336
No. 17	0.53	70	−0.002	0.006	0.012	0.015	0.024	0.042	0.405
		110	0.003	0.000	0.027	0.049	0.103	0.140	0.425
		130	0.004	0.016	0.045	0.129	0.182	0.222	0.456
No. 18	0.54	70	0.004	0.020	0.053	0.080	0.113	0.145	0.468
		110	0.013	0.109	0.242	0.454	0.542	0.565	0.743
		130	0.028	0.104	0.225	0.366	0.415	0.435	0.556
No. 19	0.55	70	−0.001	0.020	0.103	0.153	0.188	0.220	0.467
		110	0.018	0.122	0.254	0.463	0.610	0.682	0.790
		130	0.021	0.122	0.136	0.289	0.380	0.435	0.578
No. 20	0.56	70	0.004	0.082	0.236	0.388	0.459	0.479	0.686
		110	0.029	0.181	0.337	0.459	0.542	0.599	0.644
		130	0.039	0.168	0.209	0.364	0.429	0.468	0.533
No 21	0.57	70	0.001	0.007	0.019	0.023	0.034	0.048	0.247
		110	0.015	0.038	0.129	0.186	0.249	0.265	0.437
		130	0.023	0.151	0.390	0.557	0.613	0.623	0.648
No. 22	0.57	70	0.005	0.039	0.210	0.236	0.325	0.351	0.574
		110	0.015	0.112	0.288	0.425	0.555	0.563	0.702
		130	0.023	0.209	0.383	0.441	0.470	0.473	0.549
No. 23	0.68	70	−0.003	0.025	0.115	0.172	0.220	0.269	0.596
		110	0.096	0.409	0.570	0.651	0.680	0.721	0.807
		130	0.037	0.403	0.650	0.686	0.700	0.738	0.783
No. 24	0.84	70	0.100	0.277	0.343	0.373	0.393	0.427	0.513
		110	0.148	0.233	0.258	0.274	0.275	0.275	0.287
		130	0.101	0.159	0.168	0.201	0.202	0.201	0.206
No. 25	0.92	70	0.131	0.349	0.428	0.499	0.489	0.495	0.508
		110	0.234	0.322	0.351	0.360	0.360	0.353	0.356
		130	0.207	0.256	0.278	0.294	0.299	0.294	0.293
No. 26	0.92	70	0.273	0.415	0.448	0.463	0.485	0.491	0.531
		110	0.291	0.354	0.364	0.362	0.361	0.358	0.391
		130	0.302	0.382	0.385	0.386	0.384	0.392	0.399
No. 27	1.05	70	0.100	0.287	0.353	0.406	0.459	0.490	0.629
		110	0.374	0.561	0.577	0.597	0.601	0.603	0.620
		130	0.413	0.523	0.548	0.563	0.566	0.567	0.567


12 months
6 months
Cement No.
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
Expansion, per cent
0.800
0.600
0.400
0.200
0
0.971
0.916
0.901
0.861
130 F.
110 F.
70 F.
Same brand as 2 but treated with Vinsol Resin
Same brand as 12 but treated with Vinsol Resin
Same brand as 17 but treated with Vinsol Resin

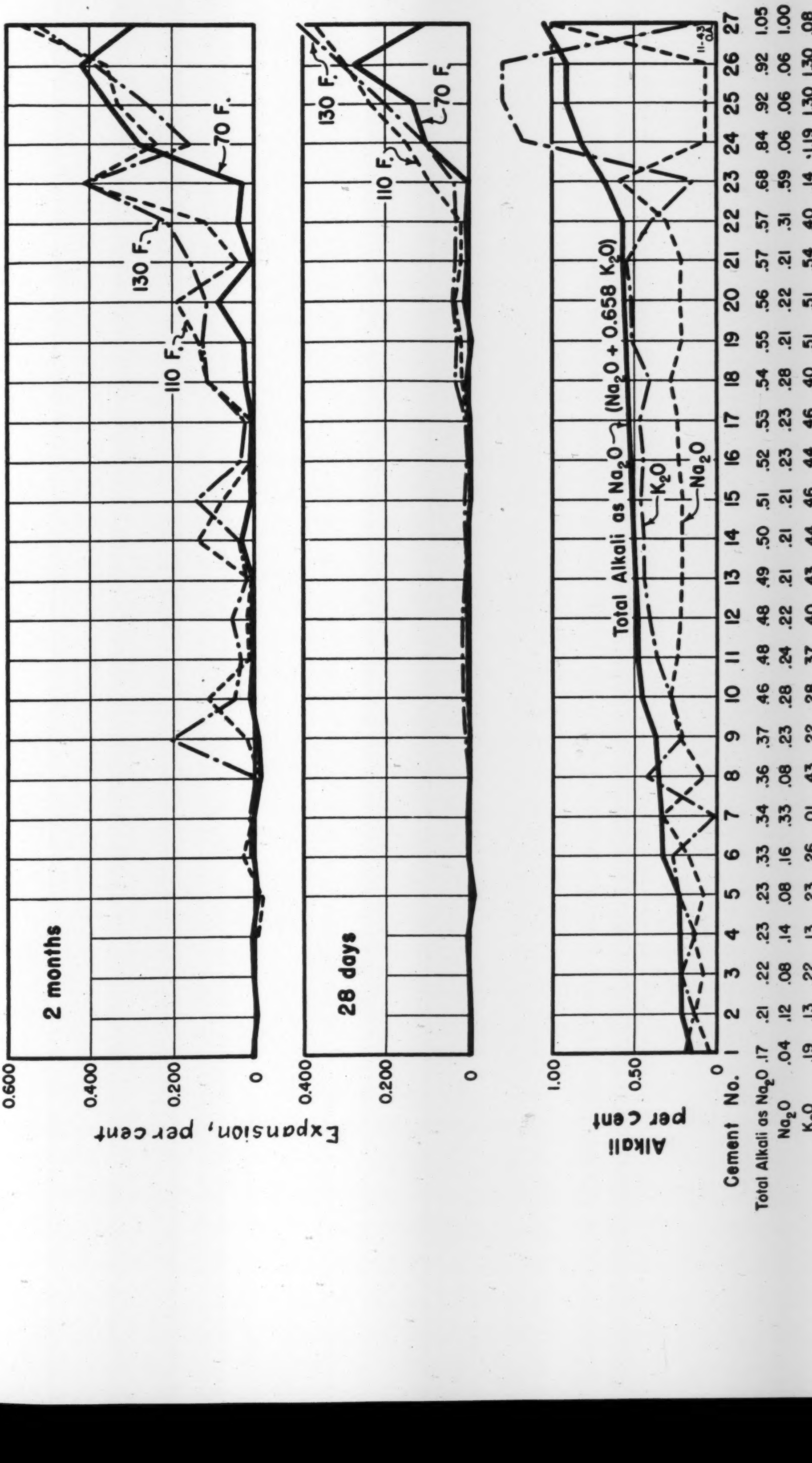
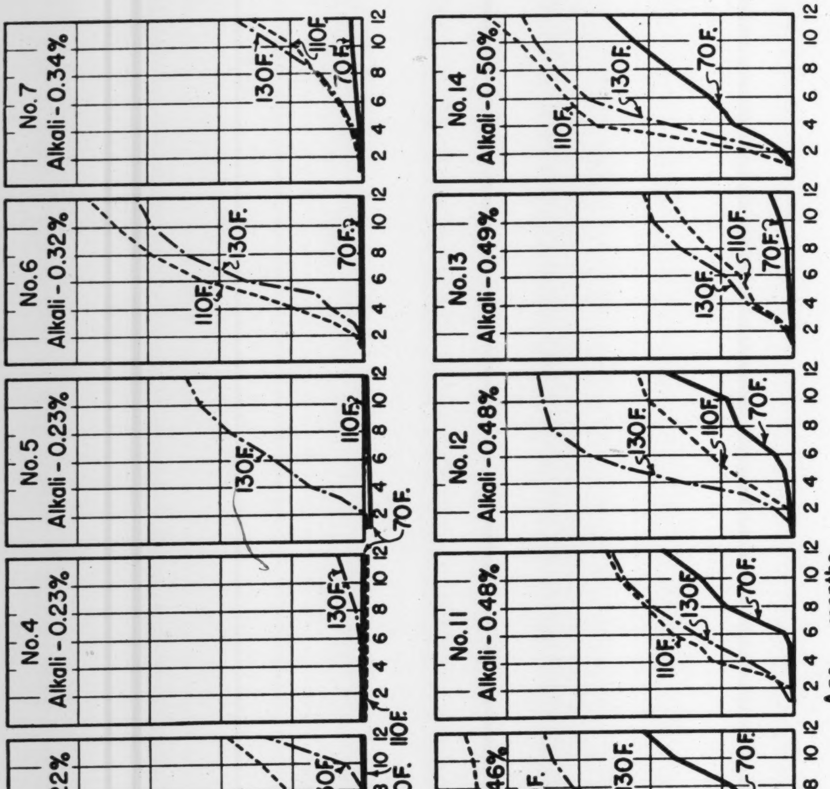


FIG. 3.—28 Day to 12 Month Expansions of 1:2 Mortars of a Neutral Sand (Perkins) Plus 10 per cent Reactive Aggregate (No. 28039) in Combination with 27 Cements from Different Sections of the United States.


No.1
Alkali-0.17%
130F.
110F.
70F.
No.2
Alkali-0.21%
130F.
70F.
110F.
No.3
Alkali-0.22%
110F.
130F.
70F.
110F.
No.4
Alkali-0.23%
130F.
110F.
No.5
Alkali-0.23%
130F.
110F.
70F.
No.6
Alkali-0.32%
110F.
130F.
70F.
No.7
Alkali-0.34%
130F.
110F.
70F.
No.8
Alkali-0.36%
130F.
70F.
110F.
No.9
Alk.-0.37%
130F.
110F.
70F.
No.10
Alk.-0.46%
110F.
130F.
70F.
No.11
Alkali-0.48%
110F.
130F.
70F.
No.12
Alkali-0.48%
130F.
110F.
70F.
No.13
Alkali-0.49%
130F.
110F.
70F.
No.14
Alkali-0.50%
110F.
130F.
70F.
1.000
0.800
0.600
0.400
0.200
0
Expansion, per cent
2
4
6
8
10
12
Age, months

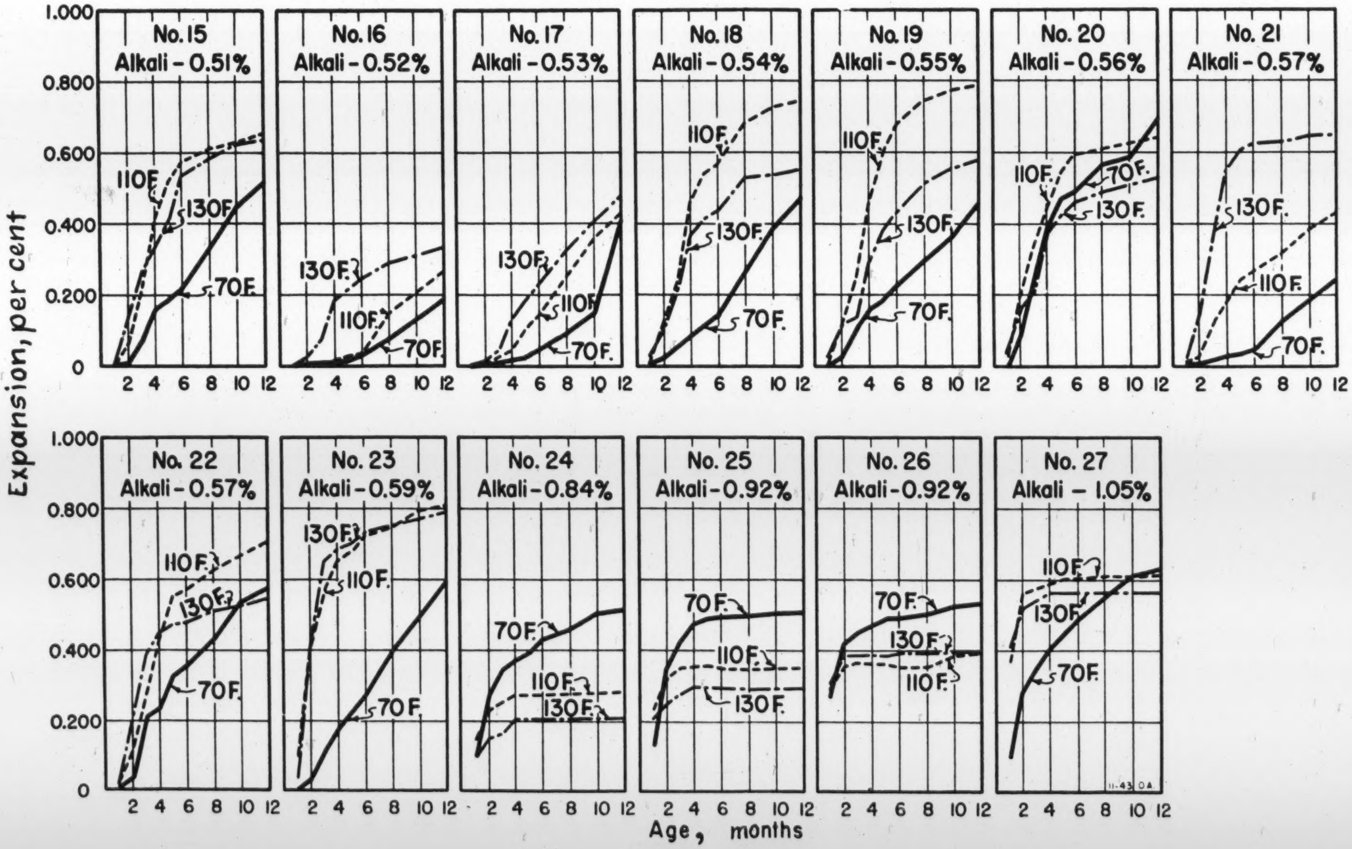


FIG. 4.—28 Day to 12 Month Expansions of 1:2 Mortars of a Neutral Sand (Perkins) Plus 10 per cent Reactive Aggregate (No. 28039) in Combination with 27 Cements from Different Sections of the United States.

It has been contended by some that higher early expansions occur at 100 to 110 F. than at temperatures either materially lower or higher, such as at 70 F. and 130 F., also that the ultimate expansion at 70 F. is greater than at 110 F. or 130 F. An opportunity was had recently to test this theory in a special study involving different type cements from scattered plants throughout the United States. The tests in this series up to 12 months are shown in Table III and Figs. 3 and 4. They are arranged in the order of increasing total alkali in the cement.

It will be noted that, as found in other California studies, the magnitude of the expansions at normal temperature is in general proportional at all ages to the alkali content of the cement. Expansions at the higher temperatures (110 and 130 F.) are quite erratic and in three or four cases appear to be decidedly out of line with what might be expected from the alkali content and the expansion at 70 F.

Particular attention is called to the high expansions at 110 and 130 F. in the case of cement No. 6, which is low in alkali (0.33 per cent) and has a low-expansion (60 units) at 6 months at 70 F., also to cement No. 3 (0.22 per cent alkali) which show no expansion in 6 months at 70 and 130 F., but moderately high expansions at 110 F., whereas cement No. 5 (0.23 per cent alkali) expanded 0.24 per cent in 6 months at 130 F. but none at all at either 70 or 110 F. Similar experience has been had with other low-alkali cement-reactive aggregate combinations, thereby indicating that reactions develop in the laboratory under constantly controlled humidity curing at 110 F. and 130 F. which never develop at 70 F., and which probably never develop in the field where temperatures as high as 130 F. or even 110 F. are a rare, or at most an infrequent occurrence.

Reproducibility of Tests:

In Table IV will be found the results of a study to determine the reproducibility of results by repeat tests on presumably the same reactive combination of cement and aggregate from which duplicate sets of specimens were cast on different days. This study served a dual purpose as it afforded an opportunity at the same time to check other studies of the relative reactivity at 70, 110, and 130 F.

Erratic behavior of reactive combinations has frequently been noted. The reasons why low-alkali cement-reactive aggregate combinations occasionally develop excessive expansion (usually at 110 or 130 F.) is at present obscure although it appears reasonable to suspect alkali in the aggregates. However, tests on thousands of specimens of all combinations during the past three or four years have shown that the over-all picture is fairly constant and that duplicate tests on identical combinations can be expected to produce the same results within reasonable limits.

The data shown in Table IV checks this conclusion. Each entry represents the average of tests on three specimens; three in each of three sets fabricated on three different days. The over-all agreement is good and checks with expected performance.

Discrepancies are probably due to any one or all of the following causes:

1. Lack of absolute uniformity in composition of ingredients.

2. Nonuniformity in fabrication procedure.

3. Variations in curing conditions such as variation in moisture in the specimens that were all cured in sealed containers in which it was difficult to maintain absolutely uniform moisture conditions in all parts of the specimen, particularly when maintained at 110 and 130 F.

With the more reactive combination,

using reactive mineral No. 28039, the pickup in expansion at 70 F. was rapid. At 12 months the expansion at 70 F. with both aggregates was close to and in some cases even exceeded that at the higher temperatures.

In earlier tests on a similar combination, the expansion at 70 F. has exceeded that at 130 F. in the even shorter period of 3 months.

TABLE IV.—EXPANSION AND MODULUS OF ELASTICITY TESTS ON THREE SETS OF SPECIMENS, WITH HIGH- AND MEDIUM LOW-ALKALI CEMENTS AND TWO REACTIVE FINE AGGREGATES

Specimens in each set fabricated on different days for the purpose of studying reproducibility of test results.
Each entry represents the average of three specimens.
1 by 1 by 10-in. bars—1:2 mix—expansion in per cent.

	Test Period	Cement G (1.07% Na_2O)						Cement A (0.41% Na_2O)					
		70 F.		110 F.		130 F.		70 F.		110 F.		130 F.	
		Exp.	M.E.	Exp.	M.E.	Exp.	M.E.	Exp.	M.E.	Exp.	M.E.	Exp.	M.E.
	SATICOY SAND												
Set No. 1	28 days	0.002	4.23	0.019	3.64	0.154	3.23	−0.005	4.44	−0.009	4.52	−0.001	4.72
	2 months	0.019		0.185		0.316		−0.002		−0.001		0.001	
	3 months	0.079	3.96	0.260	2.75	0.362	2.62	0.001	4.71	0.002	4.81	0.004	4.78
	4 months	0.136		0.314		0.391		0.000		0.012		0.001	
	5 months	0.216		0.346		0.403		−0.001		0.010		0.001	
	6 months	0.268	3.67	0.374	2.61	0.427	2.24	0.000	4.97	0.006	4.95	0.002	4.71
	8 months	0.325		0.415		0.429		−0.002		0.015		0.001	
	12 months	0.391	3.61	0.429	2.72	0.453	1.76	−0.004	5.15	0.032	4.58	0.004	4.06
Set No. 2	28 days	0.005	4.26	0.007	3.83	0.143	3.09	0.001	4.60	−0.005	4.49	0.004	4.60
	2 months	0.048		0.198		0.279		0.005		0.002		0.010	
	3 months	0.149	3.69	0.276	3.19	0.367	2.35	0.005	4.91	0.004	4.91	0.013	4.50
	4 months	0.226		0.347		0.411		0.006		0.006		0.020	
	5 months	0.274		0.405		0.441		0.005		0.008		0.029	
	6 months	0.327	3.62	0.447	3.00	0.467	1.94	0.005	5.06	0.006	4.92	0.034	4.06
	8 months	0.365		0.479		0.494		0.005		0.029		0.076	
	12 months	0.455	3.26	0.490	2.85	0.512	2.10	0.002	5.31	0.097	3.74	0.190	2.83
Set No. 3	28 days	0.003	4.20	0.022	3.60	0.123	3.57	0.000	4.57	−0.004	4.32	−0.004	4.51
	2 months	0.019		0.153		0.260		0.001		0.001		0.003	
	3 months	0.120	3.67	0.259	3.12	0.302	2.79	0.005	4.87	0.003	4.46	0.006	4.50
	4 months	0.207		0.305		0.351		0.004		0.009		0.006	
	5 months	0.287		0.361		0.367		0.003		0.007		0.009	
	6 months	0.343	3.45	0.385	3.01	0.392	2.40	0.004	5.20	0.008	4.56	0.027	3.90
	8 months	0.413		0.429		0.421		0.004		0.022		0.079	
	12 months	0.420	3.51	0.466	2.89	0.511	2.04	0.000	5.22	0.039	4.32	0.165	2.61
	RUSSIAN RIVER SAND + 10 PER CENT SILICEOUS MAGNESIAN LIMESTONE NO. 28039												
Set No. 1	28 days	0.089	3.49	0.190	2.40	0.235	2.91	−0.011	4.53	−0.017	4.40	−0.005	4.52
	2 months	0.318		0.421		0.622		−0.008		−0.009		−0.002	
	3 months	0.394	2.79	0.567	1.71	0.644	1.81	−0.004	4.80	−0.010	4.61	−0.002	4.67
	4 months	0.527		0.630		0.651		−0.008		−0.005		−0.003	
	5 months	0.581		0.680		0.655		−0.009		−0.001		−0.002	
	6 months	0.620	2.65	0.687	2.18	0.659	2.33	−0.006	5.06	−0.005	4.97	−0.003	4.90
	8 months	0.632		0.696		0.656		−0.008		0.005		−0.004	
	12 months	0.665	3.27	0.699	2.85	0.664	2.31	−0.013	5.17	0.030	4.64	+0.016	4.51
Set No. 2	28 days	0.150	3.29	0.157	2.70	0.327	2.28	−0.006	4.42	−0.013	4.54	−0.003	4.62
	2 months	0.358		0.430		0.615		−0.003		−0.010		0.002	
	3 months	0.526	2.28	0.604	1.66	0.719	1.87	−0.002	4.71	−0.006	4.75	0.002	4.71
	4 months	0.609		0.713		0.755		−0.002		−0.005		0.004	
	5 months	0.652		0.735		0.776		−0.003		−0.001		0.004	
	6 months	0.686	2.76	0.738	2.41	0.812	2.24	−0.003	5.00	−0.003	4.98	0.007	
	8 months	0.726		0.758		0.826		−0.003		0.019		0.015	4.75
	12 months	0.762	3.48	0.763	3.40	0.800	2.87	−0.007	5.31	0.083	3.89	0.121	4.48
Set No. 3	28 days	0.054	3.75	0.204	2.51	0.256	2.69	−0.007	4.48	−0.012	4.53	−0.003	4.51
	2 months	0.235		0.503		0.528		−0.005		−0.009		0.001	
	3 months	0.410	2.59	0.630	1.70	0.598	1.90	−0.003	4.85	−0.005	4.87	0.002	4.75
	4 months	0.530		0.664		0.607		−0.003		0.003		0.001	
	5 months	0.591		0.679		0.607		−0.002		−0.003		0.000	
	6 months	0.623	2.60	0.685	2.28	0.608	2.58	−0.002	4.93	−0.002	4.98	−0.002	4.58
	8 months	0.660		0.694		0.612		−0.001		0.001		0.000	
	12 months	0.687	3.35	0.677	2.81	0.612	3.06	0.002	5.08	−0.002	5.09	0.003	4.60

Curing in a Caustic Solution:

Search has been made for some modified curing procedure to avoid some of the inconsistencies which arise in the attempt to maintain a constant optimum moisture in all specimens and at the same time to accelerate the reaction.

Limited studies have been made along this line by curing in 1.0 and 2.0 per cent sodium hydroxide solutions. In Fig. 5 will be found the results of a few such tests at 70 and 110 F. It will be noted that, in general, the expansions with the known reactive sands are appreciably accelerated by the salt solutions, whereas, the nonreactive sands were not similarly affected.

However, while accelerated tests in sealed containers at 110 F. and in sodium hydroxide solutions at 70 and 110 F. have definite possibilities, further work should be done to correlate these accelerated test procedures with field experience before they can be considered as acceptable or preferred methods for the rapid detection of potentially reactive cement-aggregate combinations.

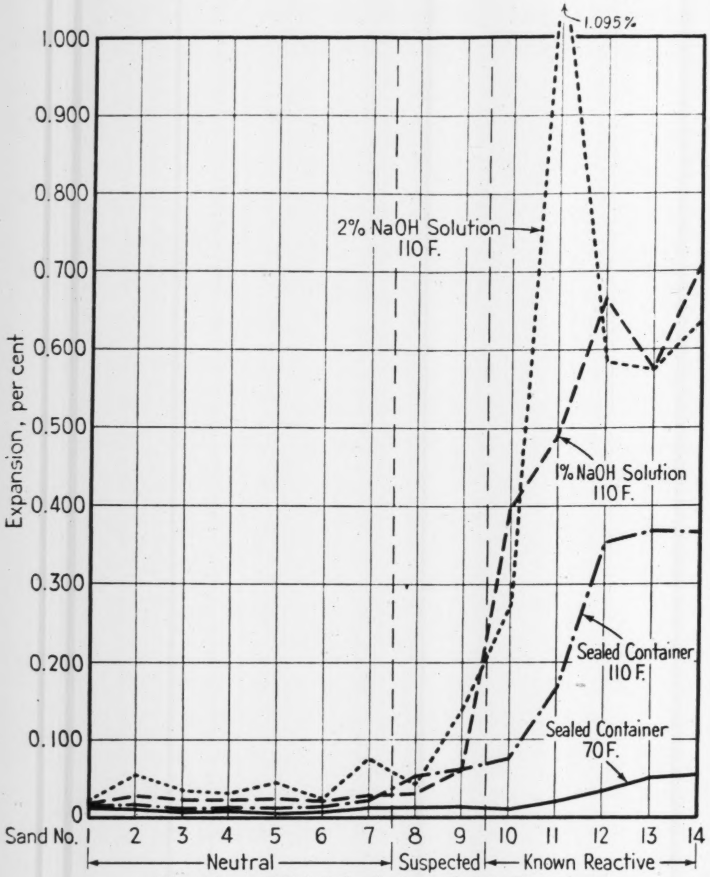


FIG. 5.—Comparative Expansion in 90 Days of 1:2 Mortars of a High Alkali Cement (1.14 per cent) and Fourteen Sands Ranging from Good to Bad Job Experience. Curing procedure as indicated.

With the fine aggregates under test, however, the evidence appears conclusive that tests in sodium hydroxide solutions, particularly at 110 F., have proven merit for quickly detecting a potentially reactive combination.

CONCLUSIONS

In the light of our present knowledge of the subject, the following conclusions are believed justified:

1. The best proven procedure for acceleration of reaction between cement and aggregate is curing under constant humidity conditions with neither excess or deficiency of the optimum moisture to accomplish the reaction.

2. The initial reaction is accelerated by temperature. Although in some cases the ultimate magnitude of the expansion may be less under higher than normal temperatures, tests at 100 to 110 F. and possibly even at 130 F. are valuable as early indications of adverse combinations. Curing at either 100 or 110 F. appears preferable to 130 F.

3. The cement content is an important factor. While no evidence has been developed to show that the excessive expansion noted in 1:2 mortars will in time be duplicated in the field with leaner mixes, nevertheless if early information is desired with regard to the probable reaction characteristics of a given fine aggregate, a 1:2 mortar mix should be used. Richer mixes should be used only where richer mixes are to be used in construction.

4. Curing of expansion test bars by immersion in an alkali solution undoubtedly accelerates the action. This test merits further study and serious consideration as a possible substitute for curing in a sealed container.

Acknowledgments:

The investigations and tests described in this paper were conducted as a research project of the California Division of Highways of which State Highway Engineer G. T. McCoy is Chief. The author is indebted to the following members of his staff who have contributed valuable suggestions: Allen Nicol, Mineral Technologist for the petrographic and fluorescence studies and for the development of the pat test in a caustic solution for rapid detection of reactive particles in the aggregate and the test for expansion in a similar solution; and to Assistant Physical Testing Engineer W. E. Haskell for supervision of fabrication and test of specimens and analyses of test data.

BIBLIOGRAPHY

For a complete bibliography of material published to date on the effect of alkalies in portland cement on concrete durability see Bibliography compiled by R. F. Blanks and published as an Appendix to the Report of Committee C-1.[6]

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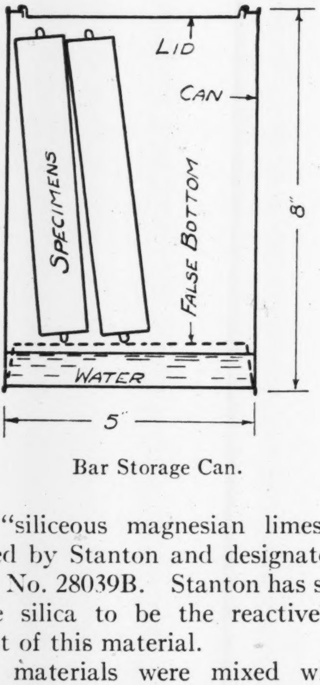
[6] See p. 219.

DISCUSSION

Mr. W. C. Hanna[1] (*presented in written form*).—It has been suggested that the results to which reference was made in our contribution to the "Notes on the Effect of Alkalies in Portland Cement on the Durability of Concrete"[2] be reported, and this is being done in discussion of Mr. Stanton's paper as being of interest to others who are working on this complex problem. It is acknowledged that a change in any one or combination of variables would be apt to give results which would produce either more or less expansions than those reported. Data obtained on the expansion of mortar bars made with reactive aggregate and 132 commercial cements furnished by Dalton G. Miller, of the U. S. Soil Conservation Service, Division of Drainage Laboratory, St. Paul, Minnesota, are presented. Also reported are some data on the effect of certain admixtures to cements of varying alkali content.

The experimental procedure was essentially the same as that described by Stanton in a paper presented before the American Society of Civil Engineers.[3] The molds and length comparator were as described in A.S.T.M. Tentative Method of Test for Autoclave Expansion of Portland Cement (C 151 - 40 T),[4] except that the gage length was 5 in. The storage cans were fitted with perforated false bottoms to hold the specimens above the water in the bottom.

The mortars consisted of one part (by weight) of cement, 2.2 parts of sand, and well water of high purity (16 ppm. sodium ion, 5.6 ppm. potassium ion) to make a consistency approximately equivalent to that described by Federal Specification SS-C-158a, Section F-4m(4)—usually 0.50 to 0.55 times the weight of the dry cement. The sand was 9 parts of a local commercial plastering sand of known good service record and 1 part of 30 to 80 mesh "siliceous magnesian limestone" supplied by Stanton and designated by him as No. 28039B. Stanton has shown opaline silica to be the reactive constituent of this material.



Bar Storage Can.

The materials were mixed with a trowel on a smooth, nonporous surface for 1.5 min. and molded as indicated in A.S.T.M. Method C 151, Section 4(*c*) and 4(*d*). The bars were removed from the molds 20 to 24 hr. after molding, measured, and stored in sealed cans in a room the average temperature of which was maintained at 70 F. with variations of from 5 to 10 F. for short intervals.

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[1] Chief Chemist, California Portland Cement Co., Colton, Calif.

[2] See p. 199.

[3] T. E. Stanton, "Expansion of Concrete Through Reaction Between Cement and Aggregate," *Transactions*, Am. Soc. Civil Engrs., Vol. 107, p. 53 (1942).

[4] 1942 Book of A.S.T.M. Standards, Part II, p. 1077.

The reactivity of the plastering sand and the opaline material was checked by preparing mortar bars of plastering sand and of Ottawa sand both with and without the opaline addition. The expansion at 2 and 18 months are given in the accompanying Table I and indicated that the plastering sand may be considered sound and that the opaline material is exceedingly reactive in the proportion used.

The commercial cements tested were 132 cements furnished by Dalton G. Miller. The results of the Medicine Lake and laboratory tests of sulfate resistance as well as chemical analyses and fineness data for 106 of these have been reported by Miller and Manson.[5] An enlarged series of other tests are being tabulated by Miller. The percentages of Na_2O, K_2O, and of total alkali calculated as $Na_2O + 0.658\ K_2O$ as furnished to us by Miller are reported in the accompanying Table II for the 26 cements not included in Miller and Manson's paper.

TABLE I.—EXPANSION OF MORTAR BARS.

Lot	Alkali in Cement, per cent	Sand	Opaline Material, per cent	Expansion, per cent	
				2 months	18 months
No. 3165	1.01	Local	None	0.012	0.034
No. 3166	1.01	Local	10	0.92	1.40
No. 3167	1.01	Ottawa	None	0.014	0.030
No. 3168	1.01	Ottawa	10	0.69	1.00
No. 3169	0.56	Local	None	0.006	0.046
No. 3170	0.56	Local	10	0.076	1.62
No. 3171	0.56	Ottawa	None	0.002	0.032
No. 3172	0.56	Ottawa	10	0.074	1.45

The expansion of the mortar bars at 2 months, 6 months, and 18 months for the standard and modified cements is shown in the accompanying Figs. 1*A*, *B*, and *C*, respectively, and the alkali content in Fig. 1*D*. The cements are arranged in order of increasing alkali content calculated as Na_2O. In Figs. 1*A*, *B*, and *C* the superimposed curves, designed to show the trend of expansion *versus* alkali content, were established by the method of least squares using second degree equations.

TABLE II.—ALKALI CONTENT OF ADDITIONAL CEMENTS FURNISHED BY D. G. MILLER.

Cement	Type	Na_2O, per cent	K_2O, per cent	Total Alkali as Na_2O, per cent
No. 659	Standard	0.04	0.16	0.14
No. 653		0.04	0.26	0.21
No. 681		0.09	0.33	0.31
No. 670		0.18	0.37	0.42
No. 682		0.18	0.40	0.44
No. 671		0.24	0.32	0.45
No. 647		0.21	0.37	0.45
No. 646		0.22	0.43	0.50
No. 665	Sulfate resisting	0.28	0.39	0.54
No. 667	Standard	0.21	0.52	0.55
No. 643		0.38	0.42	0.66
No. 683		0.26	0.65	0.69
No. 664	Sulfate resisting	0.36	0.60	0.75
No. 687	Aluminous	0.07	0.06	0.11
No. 674		0.11	0.16	0.21
No. 613		0.11	0.18	0.23
No. 637	High silica	0.12	0.35	0.35
No. 589		0.40	0.36	0.64
No. 586		0.47	0.44	0.76
No. 678		0.17	1.1	0.89
No. 177		0.52	0.57	0.90
No. 684		0.23	1.72	1.36
No. 592		1.4	0.34	1.62
No. 679	High early strength	0.15	0.39	0.41
No. 666		0.31	0.23	0.46
No. 642		0.24	0.52	0.58

It will be observed that at 2 months the trend toward increasing expansion with increasing alkali content is pronounced. At six months, although this trend is still apparent, there are a number of glaring exceptions. Twenty cements of less than 0.50 per cent alkali calculated as Na_2O have by this time attained expansion in excess of 0.1 per cent. At 18 months it is apparent that the expansion of many of the low-alkali cements

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[5] D. G. Miller and P. W. Manson, "Tests of 106 Commercial Cements for Sulfate Resistance," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 988 (1940).

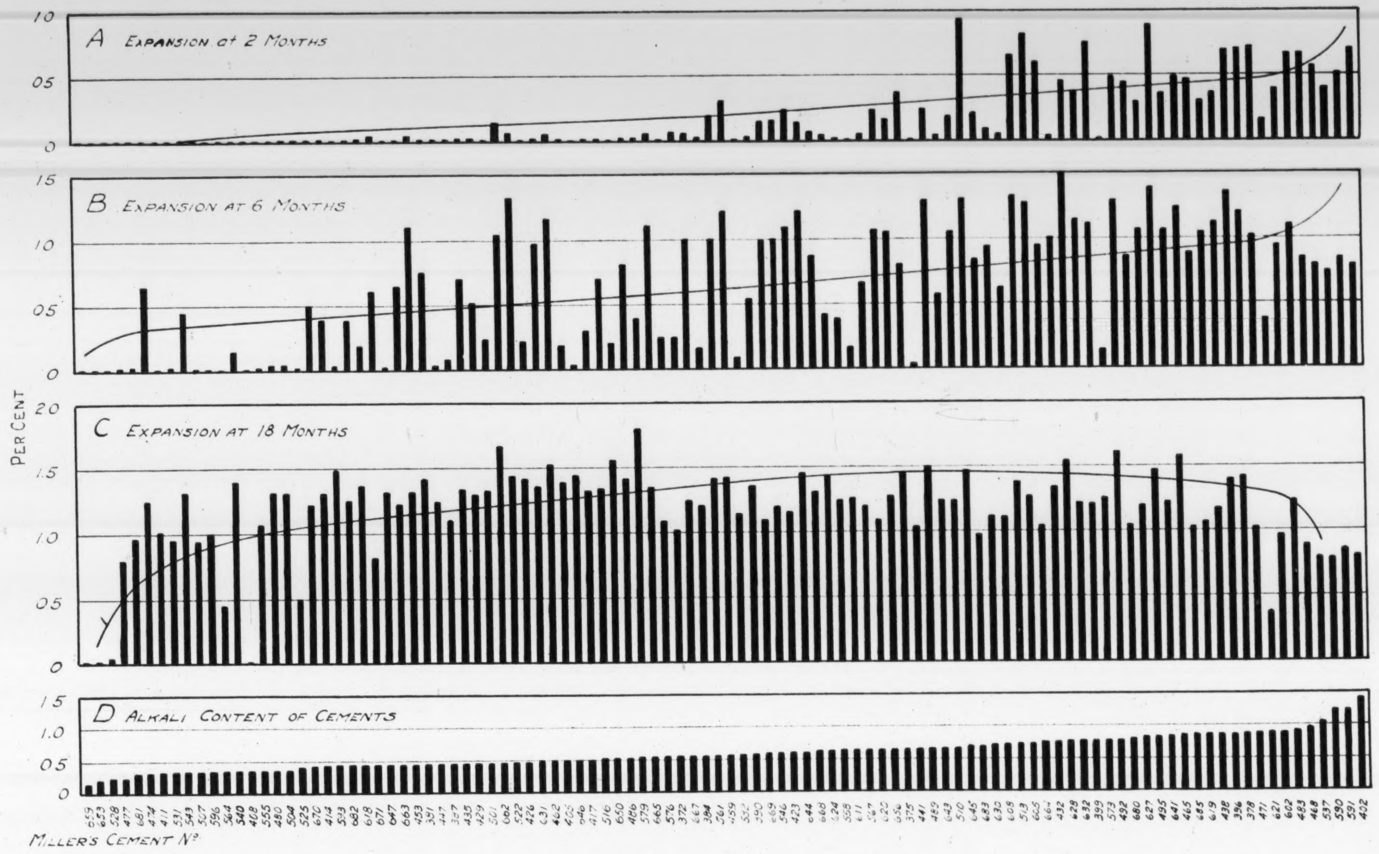

FIG. 1.—Expansion of Standard and Modified Cements.

has exceeded the expansion of many of the high-alkali cements. Two of the cements still showing low expansion at 18 months have started to expand at 22 months. These data indicate that under the conditions of test a low-alkali cement may produce as great or greater expansion with a reactive aggregate as a high-alkali cement. It should be emphasized that Stanton has shown that the magnitude of the expansion is a function of the nature, the quantity, and the particle size distribution of the reactive material.

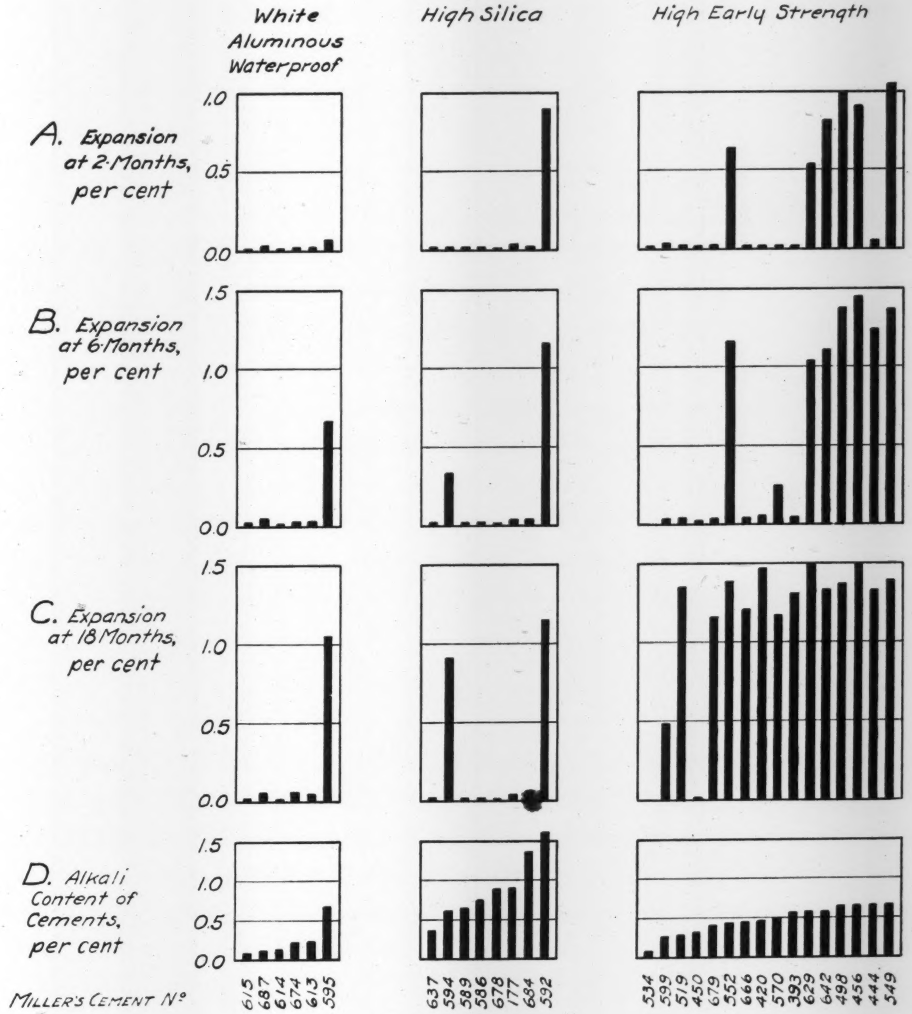

FIG. 2.—Expansion of Special Cements.

A statistical analysis of the expansion data and Miller's analytical data was undertaken to determine whether any other chemical constituent or the degree of grinding appreciably affected the expansion. Aside from a slight trend of the finer cements toward increased

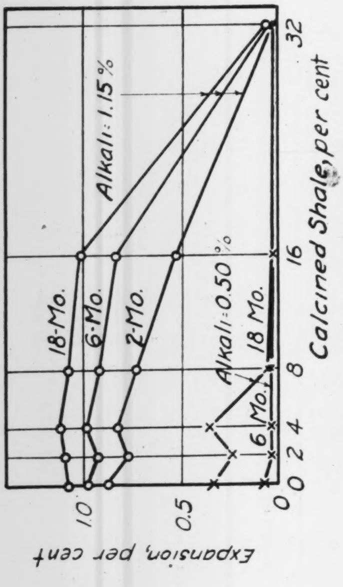

FIG. 3.—Effect of Calcined Shale.

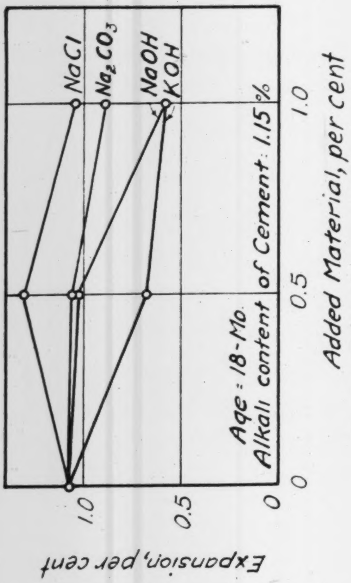

FIG. 4.—Effect of Added Alkali.

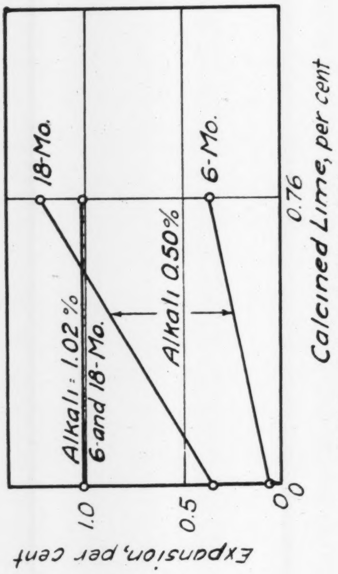

FIG. 5.—Effect of Calcined Lime.

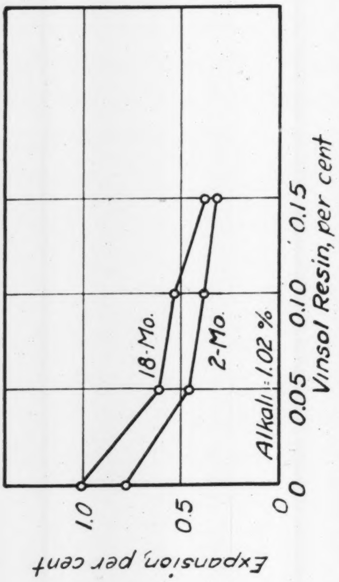

FIG. 6.—Effect of Vinsol Resin.

expansion at two months, no trend believed significant was discovered.

The expansion and alkali content data for the white, aluminous, waterproof, high-silica, and high-early-strength cements are shown in the accompanying Figs. 2*A*, *B*, *C*, and *D*. The white, waterproof, and high-early-strength cements are shown to exhibit expansion of the same order as the standard cements while the aluminous and six of the high-silica (possibly pozzuolana) cements exhibit very low expansion. This latter may be taken as confirming previous evidence that some pozzuolanic additions tend to prevent the expansion of concrete containing reactive aggregates.

The effect of varying amounts of a certain calcined shale is shown in the accompanying Fig. 3. It will be observed that additions of 2 per cent and of 4 per cent were ineffectual in preventing expansion. As much as 32 per cent appears to be required for positive prevention of expansion under the conditions of test.

The effect of alkalies deliberately added to a high-alkali cement is shown in the accompanying Fig. 4. Additions of 0.76 per cent calcined lime were also made to both high- and low-alkali cements. The expansions are shown in the accompanying Fig. 5, and it is seen that the bars made from high-alkali cement were not affected, while the expansion of the bars made from low-alkali cement were greatly increased.

Admixtures of Vinsol resin ground with the cement reduced the expansion to a limited degree as shown in the accompanying Fig. 6.

No attempt has been made to correlate the data reported here with service records, nor do the data apply to other than the specimens studied. However, the cements of the Medicine Lake series have been comprehensively investigated by Miller and others, and they include nearly every composition and type commercially available. It seems probable that under certain conditions of exposure, concretes made from reactive aggregates may eventually suffer disruptive expansion regardless of the alkali content of the cement.

Acknowledgments.—The author is indebted to Dalton G. Miller for permission to use the cements of the Medicine Lake series in this investigation and to T. E. Stanton for the reactive aggregate and the method of test. Much of the laboratory work and analysis of data was done by J. E. McKinney and Wm. F. Keyes. H. E. Kaiser contributed much helpful discussion.

MESSRS. L. SCHUMAN[6] AND F. B. HORNIBROOK[6] (*presented in written form*).—Mr. Stanton and his co-workers deserve much credit for developing such a simple and effective test method for detecting reactive combinations of cement and aggregate. The Stanton procedure, with modifications, is also followed at the National Bureau of Standards in tests for "alkali" reaction. Test specimens, consisting of 1 by 1 by 10-in. mortar bars proportioned 1:2 by weight, are kept in closed containers stored either in a 70 F. damp closet without additional water in the container, or in a 100 F. room with a small vessel containing water placed along with the bars in the container. In tests of very active materials such as opal or the California opaline limestone the aggregate for the mortar consists of standard 20–30 Ottawa sand and a small proportion of the active aggregate.

Some of the results that have been obtained and which are pertinent in a discussion of test methods are:

Early Appearance of Viscous Droplets Indicates Future Expansion.—It has been

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[6] Assistant Materials Engineer, and Materials Engineer, respectively, National Bureau of Standards, Washington, D. C.

TABLE III.—TIME AFTER MAKING 1:2 MORTAR TEST BARS, AT WHICH VISCOUS DROPLETS INDICATIVE OF ALKALI ACTION WERE FIRST NOTED (HIGH K CEMENT).

Percentage of active aggregate by weight of total aggregate	Days at 70 F.
2.5% Nevada opal, No. 30–80	2
2.5% Nevada opal, < No. 80	3
1% Nevada opal, No. 8–30	2
1% Nevada opal, No. 30–80	7
1% Nevada opal, < No. 80	3
2.5% California opaline chert < No. 80	3
20% California silica limestone, No. 30–80	5
30% Parker Dam felsites, No. 30–80	7
	Days at 100 to 105 F.
20% California silica limestone, No. 30–80	3
100% Saticoy, Calif., sand	3
100% Cowlitz River sand	3
100% Kimball, Neb., sand	5
15% Pitchstone, No. 30–80	5
15% Chalcedony, No. 30–80	5
100% Parker Dam felsites	5
100% St. Francis, Kans., sand	7
100% Cowlitz River andesite	14
100% Spokane, Wash., sand	90
100% Virginia phyllite	90 (with 1% added KOH)

noted that for test bars containing active cement-aggregate combinations, viscous droplets (probably an alkali silicate or silica gel) appear on the surface of the test bars. The accompanying Table III gives the ages at which these characteristic gel spots were first noted for test bars containing various reactive combinations. It is seen that even for reactive aggregates other than opal, potential expansion is indicated by the formation of these droplets, and that reactivity of all but the very slowest-acting materials can thus be detected in a very short time. In no case has there been abnormal expansion of the test bars which was not preceded by the appearance of the gel spots. However, there have been a few cases, as with chalcedony stored at 100 to 105 F., where scattered spots have appeared but no excessive expansion has occurred as yet. The diameters of gel spots are, in general, about the same as the maximum size of the active aggregate particles.

K_2O Has a Greater Reactivity Rate than Na_2O.—By using cements high in either K_2O or Na_2O, it has been noted that K_2O causes a greater rate of expansion than Na_2O, as may be seen in the accompanying Table IV. In all cases the expansion is greater at one month with K_2O than

TABLE IV.—COMPARISON OF RATES OF EXPANSION FOR HIGH Na AND HIGH K CEMENTS

Type of Cement	Addition	Active Material, per cent by weight of total aggregate[d]	Expansion of 1:2 Mortar at 70 F., per cent		
			1 month	3 months	6 months
High Na[a]	none	2.5% opal, No. 30-80	0.03	0.33	0.75
High K[b]	none	2.5% opal, No. 30-80	0.26	0.42	0.75
High Na	none	2.5% opal, <No. 80	0	0.01	0.06
High K	none	2.5% opal, <No. 80	0.49	1.4	disintegrated
Low alkali[c]	1.4% NaOH	2.5% opal, No. 30-80	0.08	0.08	0.23[e]
	1.4% KOH	2.5% opal, No. 30-80	0.20	0.80	1.04[e]
Low alkali	1.4% NaOH	2.5% opal, <No. 80	0.29	0.86	0.94
	1.4% KOH	2.5% opal, <No. 80	0.97	1.5	disintegrated
Low alkali	2.5% Na_2SO_4	2.5% opal, No. 30-80	0.08	0.43	0.50
	2.2% K_2SO_4	2.5% opal, No. 30-80	0.14	0.47	0.69
Low alkali	2.5% Na_2SO_4	2.5% opal, <No. 80	0.03	0.49	0.88
	2.2% K_2SO_4	2.5% opal, <No. 80	0.96	1.5	disintegrated
High Na	none	100% Parker Dam felsites	0	0.09	0.19
High K	none	100% Parker Dam felsites	0.01	0.14	0.22
High Na	none	20% Cal. siliceous limestone, No. 30-80	0.20	0.70	0.85
High K	none		0.44	0.85	1.0

[a] 1.1 per cent Na_2O, 0.02 per cent K_2O.
[b] 1.2 per cent K_2O, 0.04 per cent Na_2O.
[c] 0.14 per cent K_2O, 0.02 per cent Na_2O.
[d] Balance of aggregate standard Ottawa sand.
[e] At 5 months.

with Na_2O, and in some cases the one-month expansion with K_2O exceeds that at three months with Na_2O.

Alkali Hydroxides or Salts of Alkali Metals Can Be Added to a Low-Alkali Cement to Provide a Test Cement.—Also illustrated in Table IV is the fact that, starting with a low-alkali cement, additions of the hydroxides or salts of sodium or potassium give expansions of about the same order of magnitude as those obtained with the high-alkali cements. Tests are now under way to determine the minimum amount of each alkali which may give excessive expansion with certain aggregates.

General Discussion.—Tests with an opal from Nevada as well as with Stanton's "opaline chert" have shown maximum expansion when 5 per cent (by weight of cement) is used in a 1:2 mortar. With the high K_2O cement, the opal in the minus No. 80 size showed much more rapid expansion than in the No. 30–80 size.

A number of other aggregates have been tested. In most cases it was necessary to use the aggregate unmixed with other material, since, as with a sand from Kimball, Nebr., the percentage of opal or other active ingredient may be quite small. With some materials such as this, our petrographers (H. Insley and W. H. Parsons) have been able to pick out calcitic pieces containing opal. This opaline fraction when tested, using 40 to 50 per cent by weight of cement, shows much higher expansion than the original aggregate.

A high storage temperature (100 to 105 F.) usually, but not always, accelerates the expansion. Some materials, such as a sand from Spokane, Wash., have shown very little expansion with a high K_2O cement in one year at 70 F., but a definite, though still rather small, expansion (0.04 per cent) in 6 months at 100 to 105 F. The Kimball, Nebr., sand, however, gave greater expansion (0.07 per cent) at 70 F. than at 100 to 105 F. (0.02 per cent) in 6 months.

Though it is believed that abnormal expansions obtained with such materials as the Parker Dam felsites or the Cowlitz River, Wash., andesite are due to their glassy constituents, tests on both the natural glass obsidian and on a rhyolite containing about 50 per cent, using 30 per cent by weight of cement of the 30–80 size, show no abnormal expansion in 6 months at 100 to 105 F. However, a pitchstone tested similarly gave 0.085 per cent expansion; and silica brick (largely tridymite) gave 0.68 per cent. Tridmite silica is said to occur in some andesitic rocks.

MR. B. TREMPER[7] (*by letter*).—In Mr. Stanton's Table III are listed expansions of mortars consisting of a synthetic reactive aggregate and 27 cements as affected by the temperature of storage. Three of these cements (Nos. 2, 3, and 4) gave expansions at 6 months and earlier periods under 70 F. storage that were appreciably higher than corresponding values obtained under storage at either 110 or 130 F. These cements are exceptions to the general rule obtaining for the remaining 24.

Tests in the Washington Department of Highways' laboratory using one high-alkali cement and a synthetic reactive aggregate (from a source different from Mr. Stanton's) have consistently shown a more startling reversal of trend with temperature. As an example, specimens stored at 70 F. expanded 4000 millionths in six months, the age of Mr. Stanton's tests. When stored at 100 F. and with a slight change in testing technique the expansion at 6 months amounted to only 510 millionths.

This result is cited to call attention to a danger in using elevated temperatures of actually repressing expansion to the point that normal reactivity of an aggregate is not recognized.

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[7] Materials Engineer, Department of Highways, State of Washington, Olympia, Wash.

MR. THOMAS E. STANTON[8] *(author's closure by letter)*.—Mr. Hanna's very interesting and informative series of tests were conducted with a sand which he states is of "known" good service record. It should not be overlooked, however, that this "known" good record was based on the reaction of this sand without the addition of an active ingredient such as the opaline type of siliceous magnesian limestone which was used in the subsequent tests conducted by him. What evidence is there that this commercial plastering sand does not contain ingredients which will in time react with the known deleterious admixture in somewhat the same manner as the alkali in the cement? If such is the case, there might ultimately be an excessive expansion due to the reaction resulting from the sum of the active ingredient normally present in the sand and the alkali in the cement.

In studying this phase of the subject, it was determined that Hanna's local sand contains approximately 6 per cent alkalies (3.63 per cent Na_2O + 2.32 per cent K_2O).

It seems reasonable to assume that the ultimate excessive expansion which developed with the low alkali cements may have resulted from the later entry of the alkali in the sand into the picture. This would appear to substantiate conclusions that alkali is one of the factors at the root of the trouble, whether occuring in aggregate or the cement.

Admittedly Hanna was working with a highly reactive aggregate combination. It is significant and interesting to note that at two months, the expansions were closely proportional to the alkali content of the cement and that it was not until later that high expansion developed with most of the relatively low alkali cements.

If alkali in the aggregate is found to be a contributing factor to excessive expansion reactions, it would appear that the greater the alkali in the aggregate, the lower the permissive alkali in the cement. It is possible that erratic long time results occasionally found in the California Division of Highways tests may have been due to aklali in the aggregate. The Russian River and Perkins sands used as control sands in the California series contained 2.33 per cent Na_2O + + 0.84 per cent K_2O and 1.98 per cent Na_2O + 1.55 per cent K_2O, respectively.

In considering the erratic results found by Hanna it should not be overlooked that they represent tests on a single specimen of each combination, whereas, the California Division of Highways' tests are the average of three specimens. Frequent erratic results are encountered with one specimen in a three specimen set, which are very different from the other two specimens in the set, and which, if considered alone instead of as one of three specimens, will tend to indicate much more erratic results than have ever been observed in the California test series. Although the low expansions found by Hanna in the case of some of the relatively high alkali cements may be due to the composition of the cements, it is probable that if these cements were retested, the expansions would be found more nearly consistent with the alkali content. Where the results differ from the California Division of Highways experience, we can only conclude that all of the conditions of test were not identical.

As a concluding comment on the long time excessive expansions observed by Hanna in the case of the reactive aggregate-low alkali cement combinations, it would appear that, since combinations known to be reactive in service are invariably of reactive aggregates and high alkali cements and that these known objectionably reactive combinations develop early excessive expansions under the adopted laboratory test procedure, the long time expansions with the low alkali cements are not necessarily significant of probable field experience. There is much work yet to be done in correlating laboratory results with field performance and to this end the California Division of Highways is making a thorough periodical survey of all projects in which known reactive aggregates were used.

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[8] Materials and Research Engineer, California Division of Highways, Sacramento, Calif.

No excessive expansion cracking has been observed in any structures built during the past five years where low alkali cements were used in combination with reactive aggregates, but in the case of at least two structures where high alkali cements were used with known reactive aggregates, serious distress has already become evident; in one case being so severe as to require rather extensive protective measures to avoid failure.

TABLE V.—EXPANSION IN 12 MONTHS WITH HIGH ALKALI CEMENT (1.07 PER CENT ALKALI) AND DIFFERENT PERCENTAGES OF ADDED SODIUM HYDROXIDE. 1:2 MORTARS.

Aggregate	70 F.				130 F.			
Added NaOH, per cent	0	1	2	3	0	1	2	3
Russian River sand	0.004	0.033	0.058	0.068	0.044	0.218	0.297	0.366
Russian River sand +2.5 per cent opaline chert No. 28038	1.059	0.493	0.035	0.038	1.490	0.364	0.133	0.183

Messrs. Schuman and Hornibrook do not deem it necessary to place any free water in the container when specimens are stored in the moist closet. If this procedure insures sufficient moisture for maximum potential reaction, it obviates the objection raised by some that when specimens stored in sealed containers rest in water, the lower portion of the specimen may be contaminated by an excess of reaction products leached from the upper portion of the specimen. The answer in this case would appear to be to leave out the water.

The remarkably short period noted by the authors in which the viscous droplets of silica gel appear in the case of reactive aggregate specimens indicates a possible procedure for detecting potentially reactive aggregates which is superior to any previously advanced unless it can be considered comparable to the test in a sodium hydroxide solution.

Such tests as have been made by the California Division of Highways with high K_2O cements do not check the conclusions of Schuman and Hornibrook that K_2O causes a greater initial rate of expansion than Na_2O as will be noted by reference to Figs. 3 and 4 were, although the high K_2O cements Nos. 20, 24, 25, and 26 all developed much greater expansion in from 28 days to 2 months at 70 F. than the high Na_2O cements Nos. 23 and 27, other cements relatively high in K_2O reacted quite differently, the early rates of expansion being no greater than and sometimes less than the rates of other cements relatively high in Na_2O.

However, even though the initial rate in the case of a high K_2O cement may be greater than for the Na_2O, the ultimate magnitude of the effect of the K_2O does not appear any greater than with the Na_2O (Figs. 3 and 4).

The authors quote figures showing the effect of adding the hydroxides or salts of sodium or potassium to a low alkali cement and further report that tests are now under way to determine the minimum amount of each alkali which may give excessive expansion with certain aggregates.

In this connection, the accompanying Table V may be of interest. Tests were made on the effect of adding NaOH to a high alkali cement. It will be noted that with a nonreactive aggregate the expansions increased with increasing percentages of NaOH, whereas, with a highly reactive aggregate, the addition of 2 to 3 per cent of sodium hydroxide had the effect of reducing the expansion particularly when the tests were conducted at 70 F.

The authors found 5 per cent by weight of the cement in a 1:2 mortar to be the most reactive percentage of opaline chert (presumably No. 28038). This checks with the California studies which show 2.5 to 3.0 per cent by weight of the sand to be the optimum percentage of this high opal content rock.

Bailey Tremper has pertinently pointed out the erratic effects of temperature on the reactions. He calls attention to an extreme case where the expansion at 70 F. was 0.400 per cent in 6 months, whereas, the companion specimen stored at 100 F. expanded only 0.051 per cent

By reference to Table III and Figs. 3 and 4 it will be noted that while three of the high alkali cements (Nos. 24, 25, and 26) developed markedly greater early as well as subsequent expansion at 70 F. than at either 110 or 130 F. with most of the cements in this series the expansion at temperatures of 110 and 130 F. were greater than at 70 F. Furthermore, there was no consistency; 110 F. being more effective with some cements and 130 F. with others. In some cases, temperature of either 110 or 130 F. seemed to activate the cements abnormally. Certainly such cements should be the subject of suspicion if proposed for use in concrete construction subject to above normal temperatures for extended periods of time.

A STUDY OF THE HEAT OF SOLUTION PROCEDURE FOR DETERMINING THE HEAT OF HYDRATION OF PORTLAND CEMENT

By Leo Shartsis[1] and Edwin S. Newman[1]

Synopsis

The reproducibility of the method of measuring heats of hydration of portland cement described in Federal Specification for Cements, Hydraulic: General Specifications (Methods for Sampling, Inspection, and Testing) (SS-C-158a) was investigated by the study of data accumulated in routine testing. The data obtained by a slightly modified method were also studied to determine their precision.

The isothermal heat of solution of zinc oxide was determined with a precision calorimeter in the concentrations of acid prescribed in the Federal Specification and found to be 256.1 cal. per g., the value given by the specification at 30 C., with a temperature coefficient of −0.1 cal. per deg. Cent. per g. in the range 20 to 35 C.

The temperature coefficients of the heats of solution and hydration of a cement were found to be −0.20 and +0.1 cal. per deg. Cent. per g., respectively, in the range 20 to 30 C.

Changes in the present procedure for determining the heat of hydration of portland cement are suggested and discussed.

The heat-of-solution method for determining the heat of hydration of portland cement has been required by Federal Specification for Cement; Portland, Moderate-Heat-of-Hardening (SS-C-206) for several years and has been used in the acceptance testing of large quantities of cement. The only data available, however, on the precision of the type of calorimeter specified are the few data presented by Lerch (**1**).[2] The present paper (*a*) presents the results of a more complete investigation of the precision of this calorimeter, (*b*) supplies data needed to obtain increased accuracy, and (*c*) suggests means by which the precision and accuracy of the method described in Federal Specification SS-C-158a may be improved.

Materials, Apparatus, and Procedure

Data were obtained from two sources. One was an accumulation of the results of acceptance tests. Some of these determinations were made to verify heats of hydration which had been found to exceed the value allowed by the specification and which were repeated before rejecting the cement. Other repeated tests were made on cements which were very close to the specification limits for heat of hydration, while additional duplicate measurements were made occasionally as a part of the routine to test for the absence of errors in manipulation. The acceptance tests were performed between June, 1940, and January, 1941, inclusive, and are represented in this study by 85 determinations of heat capacity, 98 determinations of the heat of solution of dry cements, and 44 and 53 determinations of the heat of solution of the hydrated paste at 7 and 28 days, respectively.

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[1] National Bureau of Standards, Washington, D. C.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 916.

The remainder of the data were obtained as the result of an investigation of the reproducibility of heat-of-hydration measurements made by a slight modification of the method described in the Federal Specification SS-C-158a. Five representative cements were selected for the latter investigation. These were two low-heat, two moderate-heat, and one standard portland cement. Each lot of cement was mixed for several hours in a jar mill with the aid of a few wooden cubes to insure uniformity. The cements were then sealed in mason jars and stored at 70 F. (21 C.) Six samples of paste were prepared from each cement in accordance with the methods given in Federal Specification SS-C-158a, except that the initial temperature of the cement and water was 70 ± 3 F. (21 ± 1.7 C.) instead of being adjusted to such a temperature that the paste after mixing for 5 min. was at 70 ± 5 F. (21 ± 2.8 C.) One sample of paste was prepared each day and stored in four sealed vials until tested. Immediately after sealing, two of these vials were agitated for 1 hr. by turning them end-over-end at about 22 rpm. The heats of solution of the samples of hardened cement paste, both agitated and unagitated, were determined at the age of 7 days and again at 28 days. On each of those days and in the same calorimeter, the heat of solution of the corresponding dry cement was determined.

A detailed drawing and a description of the Dewar flask calorimeter used for the determination of the heat of hydration of cement are given in the Federal

TABLE I.—HEATS OF SOLUTION AND HYDRATION OF PORTLAND CEMENTS.

NOTE.—The heats of solution and hydration in this table are calculated in exact accordance with the Federal Specification SS-C-158a. However, the temperature of the cement and water before mixing was 70 ±3 F. instead of being adjusted to such a temperature that the paste after mixing was at 70 ±5 F. Column (*A*) in the table presents the heats of hydration at 28 days, calculated according to the specification, and column (*B*) shows the values obtained by subtracting the heat of solution of the 28-day-hydrated cement from that of the dry cement determined on the same day.

Cement[a]		7-Day Period			28-Day Period			
		Heat of Solution, cal. per g.		Heat of Hydration, cal. per g.	Heat of Solution, cal. per g.		Heat of Hydration, cal. per g.	
		Dry Cement	Hydrated Cement		Dry Cement	Hydrated Cement	(*A*)	(*B*)
No. 646		617.2	543.8	73.4	615.9	529.2	88.0	86.7
		616.6	544.3	72.3	617.3	530.6	86.0	86.7
		616.4	544.1	72.3	618.2	532.7	83.7	85.5
		617.7	543.5	74.2	617.3	531.0	86.7	86.3
		618.5	545.4	73.1	616.6	531.5	87.0	85.1
		617.9	544.5	73.4	616.7	531.2	86.7	85.5
	Avg....	617.4	544.3	73.1	617.0	531.0	86.4	86.0
	S......	0.73	0.59	0.67	0.71	1.05	1.33	0.63
	R. I.....	[53]	[73]	[53]	[67]	[40]	[47]	[53]
No. 647		592.4	538.5	53.9	591.3	523.9	68.5	67.4
		592.7	537.9	54.8	592.3	522.7	70.0	69.6
		592.0	538.5	53.5	591.4	521.7	70.3	69.7
		592.5	538.0	54.5	592.0	522.3	70.2	69.7
		590.7	537.3	53.4	591.0	522.2	68.5	68.8
		592.3	536.0[b]	56.3[b]	591.9	522.0	70.3	69.9
	Avg....	592.1	538.0	54.0	591.6	522.5	69.6	69.2
	S......	0.66	0.41	0.55	0.45	0.71	0.81	0.87
	R. I.....	[67]	[80]	[84]	[93]	[67]	[47]	[67]
No. 648		608.4	550.1	58.3	606.4	524.3	84.1	82.1
		608.2	549.8	58.4	607.9	527.6	80.6	80.3
		609.8	549 1	60.7	608.4	526.6	83.2	81.8
		608.0	548.7	59.3	608.7	528.1	79.9	80.6
		608.5	547.5[b]	61.0[b]	606.0	527.4	81.1	78.6
		608.0	550.5	57.5	608.0	527.2	80.8	80.8
	Avg....	608.5	549.6	58.8	607.6	526.9	81.6	80.7
	S......	0.62	0.66	1.09	1.01	1.24	1.50	1.14
	R. I.....	[67]	[60]	[50]	[47]	[67]	[40]	[33]
No. 649		606.3	542.6	63.7	605.9	526.3	80.0	79.6
		606.6	543.0	63.6	607.4	530.5	76.1	76.9
		607.6	541.5	66.1	607.6	528.6	79.0	79.0
		605.5	541.8	63.7	605.9	525.4	80.1	80.5
		605.2	541.1	64.1	605.7	526.9	78.3	78.8
		604.3	542.3	62.0	605.4	526.3	78.0	79.1
	Avg....	605.9	542.0	63.9	606.3	527.3	78.6	79.0
	S......	1.06	0.65	1.20	0.86	1.72	1.36	1.09
	R. I.....	[33]	[60]	[40]	[47]	[33]	[33]	[47]
No. 650		584.4	534.4	50.0	583.9	518.5	65.9	65.4
		583.7	534.5	49.2	583.3	519.1	64.6	64.2
		584.7	534.1	50.6	582.8	522.7	62.0	60.1
		583.5	533.4	50.1	582.3	520.0	63.5	62.3
		584.4	533.3	51.1	582.9	521.6	62.8	61.3
		583.2	535.7	47.5	583.0	520.8	62.4	62.2
	Avg....	584.0	534.2	49.8	583.0	520.4	63.5	62.6
	S......	0.55	0.80	1.16	0.49	1.46	1.35	1.76
	R. I.....	[73]	[47]	[47]	[87]	[27]	[27]	[27]

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[a] The standard deviation $S = \sqrt{\Sigma d^2/n}$ in which d is the deviation of the individual values from the average and n is the number of determinations.

The reproducibility index, *R. I.*, is the percentage of all the possible combinations of pairs of determinations, the individuals of which differ from each other by not more than some selected value, in this case 1 cal. per g.

[b] Bulges were found in the protective coating after the determination was completed. These values were not included in calculations of average, *S* or *R. I.*

Specification. This apparatus is a modification by Lerch (1) of that described by Pierce and Larmour (2). The speed of rotation of the stirrer was judged by the use of a stroboscope consisting of a paper disk, marked with nine equally spaced broad radial lines, glued to the top of the horizontal member of the friction drive and illuminated by the light from one electrode of a 3-watt neon lamp. This lamp was so constructed and shaded that 60 flashes of light per second were obtained when it was operated on 60-cycle alternating current. When the disk is rotated at a speed of 400 rpm. the lines, thus illuminated, appear stationary. When the speed is somewhat greater than 400 rpm. the lines appear to move in the direction of rotation, and at slower speeds they appear to move in the opposite direction.[3] A slide-wire resistance in the power supply to the motor permitted the speed to be adjusted and maintained approximately constant.

Preliminary work had shown that, in order to attain temperature equilibrium, it was desirable to assemble the calorimeter and operate the stirrer for 15 min. before introducing the sample. A solution period of 20 min., used in all determinations, was sufficiently long to insure complete dissolving of even the dry cement samples and to re-establish an approximately steady rate of transfer of heat between the calorimeter and its surroundings.

REPRODUCIBILITY OF HEATS OF SOLUTION AND HYDRATION

The heats of solution of the five cements investigated, together with the heats of solution of the unagitated pastes and the heats of hydration, are shown in Table I. The heats of solution of the agitated pastes, not included in Table I, were more scattered than those of the unagitated pastes for four out of the five cements at 7 days and for three out of five at 28 days. The root-mean-square standard deviations of the heats of solution were calculated and found to be 1.28 and 1.55 cal. per g. at 7 and 28 days, respectively, for the agitated pastes, whereas for the unagitated pastes they were 0.64 and 1.29 cal. per g. at 7 and 28 days, respectively. When broken, the hardened pastes that had been agitated showed a banded structure which indicated that segregation had occurred before hardening had taken place.

In Table I are given the average and the standard deviation of each series of measurements on a single cement, together with the reproducibility index, *R.I.* The standard deviation is calculated by the formula

$$S = \sqrt{\Sigma d^2/n}$$

where:

S = the standard deviation of the series of determinations,

d = the deviation of the individual measurements from the average for the series, and

n = the number of observations.

The members of a series of measurements may be combined in pairs of duplicates. The total possible combinations of pairs of determinations in the series given in Table I include a number of pairs, the individuals of which differ from each other by not more than some selected value, in this case, 1 cal. per g. The reproducibility index expresses this number as a percentage of the total possible number of combinations. Expressed in a different manner, it may be said that if the reproducibility index of a series of measurements is 60, duplicate determinations agree within the selected value, 1 cal. per g., in 60 per cent of the cases. The reproducibility index was determined by arranging the measurements in all possible combinations of pairs and determining by inspection in what percentage of the total number the difference was not greater than 1 cal. per g.

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[3] Speeds which are multiples of 400 rpm. will also cause the lines to appear stationary, but in practice no confusion arises. Four hundred rpm. is only slightly faster than speeds than can be timed by allowing a projecting set-screw to touch the finger and counting the revolutions, and no difficulty arises in distinguishing between 400 rpm. and speeds two or more times as great.

Ordinarily in the estimation of the precision of a measurement a long series of observations is made. It is possible, however, to make a fairly reliable estimate from the results of a number of short series (or pairs) of measurements, all presumably made under approximately the same conditions and, therefore, all with practically the same precision (**3**). Such an estimate has been made. Table II presents for comparison the values of σ* obtained from the results of the routine tests above described and the values obtained for the five cements later investigated. The authors invite attention to the fact that the values of σ for the routine determinations were calculated from a number of series of two or three determinations, whereas the values for the five cements later investigated were calculated from series of six determinations. Ten cements are represented by the routine samples, including four of the five cements later investigated.

TABLE II.—PRECISION OF MEASUREMENTS MADE WITH THE DEWAR FLASK CALORIMETER.

N = total number of measurements. m = number of samples or groups of measurements.

Determination	Routine Tests			Later Investigation		
	σ	N	m	σ	N	m
Heat capacity, cal. per deg. Cent.	0.60	85	22	0.43	28	9
Heats of solution, cal. per g.						
Dry cement	1.22	98	30	0.81	60	5
7-day paste	1.46	44	22	0.71	28	5
28-day paste	1.07	53	25	1.41	30	5
Heats of hydration, cal. per g.						
7-day	1.63	32	16	1.08	28	5
28-day	1.38	41	20	1.41	30	5

With the exception of the heat of solution of the hydrated cement at 28 days, the results in Table II indicate that the routine tests are the less precise. Part of the difference in precision may well have been due to the fact that the measurements on the five cements later investigated were made during the winter when the room temperature could be controlled and maintained nearly constant by the operator.

Of the heat-of-solution measurements made during the routine tests, those of the hydrated paste at 28 days were the most precise, followed in order of decreasing precision by the heats of solution of the dry cement and the hydrated paste at 7 days. The measurements during the later investigation are in the reverse order of precision, the heat of solution of the dry cement again being intermediate in precision between those of the two hydrates. This reversal is reflected in the heats of hydration. In the routine tests, the 28-day heats of hydration were the more precise while in the later investigation they were less precise than the 7-day heats of hydration. The reproducibility indices of the heats of hydration at 7 and 28 days were 33 and 39 per cent, respectively, in the routine tests. The reproducibility indices of the 7- and 28-day heats of hydration determined in the later investigation were 50 and 41 per cent, respectively.

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*σ is the standard deviation that would be obtained if an infinite number of determinations were made, that is, the standard deviation of the parent population. It is estimated by the formula:

$$\sigma = \sqrt{\frac{n_1S_1^2 + n_2S_2^2 + \cdots + n_mS_m^2}{n_1 + n_2 + \cdots + n_m - m}}$$

in which n_1, n_2, n_m are the number of measurements in each of m short series (or pairs) of determinations and S_1, S_2, S_m are the corresponding standard deviations of the short series. The sum of $n_1 + n_2 + \ldots + n_m$ is equal to the total number of measurements, N.

The average heat of solution of the 28-day dry cement samples was 0.48 cal. per g. less than that of the corresponding 7-day samples. The ratio of this difference to its standard error (4) is 2.5. A ratio of 2 or more is usually considered significant. Further evidence that this difference is real is given in Table III which shows the losses on ignition obtained at 7 and 28 days. It will be seen that for four out of five cements the average loss at 28 days was larger than at seven days, the average difference being +0.03 per cent. The ratio of this difference to its standard error is 6.7 and the difference is therefore highly significant.

TABLE III.—LOSS ON IGNITION OF PORTLAND CEMENTS.

Each value of the loss on ignition is the average of six determinations.

Cement	At 7 days		At 28 days	
	Loss on Ignition, per cent	Standard Deviation, per cent	Loss on Ignition, per cent	Standard Deviation, per cent
No. 646	1.12	0.012	1.15	0.013
No. 647	1.43	0.024	1.46	0.015
No. 648	0.70	0.011	0.77	0.025
No. 649	0.95	0.026	0.99	0.010
No. 650	0.99	0.015	0.97	0.015

REFINEMENTS FOR IMPROVING THE ACCURACY AND PRECISION OF THE MEASUREMENTS

Temperature Coefficients of the Heats of Solution and Hydration:

The heats of solution of zinc oxide, of dry and of hydrated cement, the heat of hydration of cement, and the heat capacity of the calorimeter, are all functions of temperature. The temperature coefficients of these properties were therefore either measured or derived from data in the literature. Furthermore, since the value for the heat of solution of zinc oxide (5) given in the Federal Specification was obtained in a solution where the concentrations of the reactants were not those prescribed by the specification, it was desirable to determine this heat of solution under the actual conditions prevailing in the tests.

The heat of solution and its temperature coefficient over the range 20 to 35 C. were determined for zinc oxide in a precision calorimeter (6), using the same concentrations of reactants (2.13 molal HNO_3, 0.6 molal HF, molar ratio $ZnO/HNO_3 = 0.109$) as those prescribed in the Federal Specification. From 17 determinations at various temperatures, it was calculated by the method of least squares that the isothermal heat of solution of zinc oxide at 30 C. was 256.1 cal. per g., with a standard deviation of the mean of 0.14 cal. per g., and that the temperature coefficient was −0.1 cal. per deg. Cent. per g. This value of the heat of solution is the same as that given in the specification, which, however, does not state the temperature at which the value 256.1 cal. per g. is correct. In a similar manner it was found that the temperature coefficient of the heat of solution of a dry cement over the range 18 to 35 C. was −0.2 cal. per deg. Cent. per g. The temperature coefficient of the heat of solution of the hydrated cement was calculated from the data herein reported combined with that of Carlson and Forbrich (7) and found to be −0.3 cal. per deg. Cent. per g. The temperature coefficient of the heat of hydration of portland cement was calculated from the heat capacity of dry cement and water (0.2 and 1.0 cal. per deg. Cent. per g., respectively) and the value (0.4 cal. per deg. Cent. per g. of ignited cement) for the hydrated paste obtained by interpolation of data given by Carlson and Forbrich. The temperature coefficient of the heat of hydration was found to be +0.1 cal. per deg. Cent.

per g. of ignited cement.[4] This value was used in calculating the heat of hydration at 25 C. from data obtained at other temperatures.

Composition of Final Solution in the Calorimeter:

It should be pointed out that, to make the determination of the heat of hydration exact, the composition of the final calorimeter solutions should be identical when the heats of solution of the dry and hydrated cements are determined. This would require the addition of 1.2 g. of water to the calorimeter at the same time as the dry cement and the inclusion of this amount of water in the heat capacity of the reactants. The effect of such an addition can be calculated approximately from the partial molal heat content of the water in 2 molal nitric acid (8), and has thus been found to be −0.15 cal. per g. of water added, or about −0.06 cal. per g. of ignited cement. This value is insignificant in this work and hence may be neglected.

TABLE IV.—COMPARISON OF ORIGINAL AND RECALCULATED[a] DATA.

Cement	7-day period						28-day period							
	Heats of Solution, cal. per g.				Heats of Hydration, cal. per g.		Heats of Solution, cal. per g.				Heats of Hydration, cal. per g.			
	Dry Cement		Hydrated Cement		Original	Recalculated	Dry Cement		Hydrated Cement		7-day Dry minus 28-day Hydrated Cement		28-day Dry minus 28-day Hydrated Cement	
	Original	Recalculated	Original	Recalculated			Original	Recalculated	Original	Recalculated	Original	Recalculated	Original	Recalculated
HEATS OF SOLUTION AND HYDRATION[b]														
No. 646	617.4	619.2	544.3	545.9	73.1	73.5	617.0	618.7	531.0	532.0	86.4	87.4	86.0	86.9
No. 647	592.1	593.6	538.0*	539.4*	54.0*	54.3*	591.6	593.6	522.5	523.5	69.6	70.2	69.2	70.0
No. 648	608.5	610.4	549.6*	551.0*	58.8*	59.6*	607.6	609.4	526.9	528.8	81.6	81.8	80.7	80.8
No. 649	605.9	607.9	542.0	543.5	63.9	64.5	606.3	607.7	527.3	528.3	78.6	79.8	79.0	79.6
No. 650	584.0	585.5	534.2	535.5	49.8	50.1	583.0	584.6	520.4	521.5	63.5	64.2	62.6	63.3
STANDARD DEVIATIONS OF HEATS OF SOLUTION AND HYDRATION[c]														
No. 646	0.73	0.46	0.59	0.76	0.67	0.65	0.71	0.86	1.05	0.82	1.33	0.88	0.63	0.47
No. 647	0.66	0.70	0.41	0.41	0.55	0.37	0.45	0.56	0.71	0.74	0.81	0.92	0.87	1.04
No. 648	0.62	0.68	0.66	0.55	1.09	0.69	1.01	0.81	1.24	0.70	1.50	1.12	1.14	1.13
No. 649	1.06	1.03	0.65	0.37	1.20	0.87	0.86	0.82	1.72	1.46	1.36	1.00	1.09	0.77
No. 650	0.55	0.56	0.80	0.70	1.16	0.94	0.49	0.53	1.46	1.70	1.35	1.42	1.76	1.81

[a] In recalculating, all corrections have been used.
[b] Each value is the average of six determinations, except those marked * which are the average of five.
[c] The standard deviation $S = \sqrt{\Sigma d^2/n}$ in which d is the deviation of the individual measurements from the average, and n is the number of determinations.

Temperature Coefficient of the Heat Capacity of the Dewar Flask Calorimeter:

The heat capacity of the acid mixture and its temperature coefficient, as distinct from those of the calorimeter itself, were calculated from measurements of the heat capacity of the precision calorimeter when filled with the acid mixture and when filled with distilled water. From heat-capacity measurements using the Dewar flask calorimeter and from the known heat capacity of the acid mixture, the heat capacity of the glass Dewar flask, stirrer, and thermometer was determined. The values 0.18 cal. per deg. Cent. per g. for the heat capacity and 0.00036 cal. per deg.^{2} per g. for the temperature coefficient of heat capacity of glass (9) were used in these calculations. From the data thus obtained the temperature coefficient of the heat capacity of the Dewar flask calorimeter and acid mixture was estimated to be +0.45 cal. per deg.[2] The heat capacity of the vacuum flask calorimeter measured at one temperature may be corrected to the temperatures at which heats of solution have actually been determined by means of this coefficient. However, it is advisable, if these temperatures differ by more than 2 or 3 deg. from that at which the heat capacity was measured, to redetermine the heat capacity of the calorimeter.

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[4] The actual water-cement ratio of the paste, based on determinations of loss on ignition, was 0.33, instead of the value 0.40 calculated from the amount of water added. The temperature coefficient of the heat of hydration is, therefore, found to be +0.13 cal. per deg. Cent. per g. of ignited cement instead of +0.17 cal. per deg. Cent. per g.

Effect of Applying Refinements:

The data thus obtained on the heat of solution of zinc oxide and the various temperature coefficients were used to recalculate the data presented in Table I. In addition the correction for the temperature of the solid reactant at the time of its introduction into the calorimeter was made in all determinations, regardless of whether or not it differed by more than 2 C. from the calorimeter temperature. The value 0.4 cal. per deg. Cent. per g. of ignited cement was taken for the specific heat of the hydrated cement paste. Table IV shows a comparison between the original and the recalculated heats of solution and hydration. The heats of solution shown are those calculated to the final temperature of the calorimeter at the completion of the determinations on the hydrated cement. All heats of hydration have been calculated to 25 C. This accounts for the occasional deviation in Table IV of the heats of hydration from those values obtained by subtracting the heats of solution of the hydrated pastes shown in the table from the values for the corresponding dry cements.

It will be noted that the recalculated heats of solution are higher in all cases. The increase is, on the average, 1.7 cal. per g. for the dry cements and 1.3 cal. per g. for the hydrates. The heats of hydration are also higher, on the average, by 0.6 cal. per g. Table V presents for comparison the values of σ obtained in the investigation of the five cements when using the specification method of calculation, together with the values obtained when all of the corrections described in the preceding paragraphs were used. It will be seen that there is a small but definite increase in precision when the corrections are applied. A large increase in the reproducibility was not to be expected in this instance since the original determinations were made under relatively favorable conditions. The room temperature did not vary greatly either during the course of a single determination or during the entire series of determinations, and in no group did the differences in the final temperatures of the calorimeter exceed 0.9 C.

TABLE V.—EFFECT OF REFINEMENTS ON THE PRECISION OF MEASUREMENTS MADE WITH THE DEWAR FLASK CALORIMETER.

N = total number of measurements. m = number of samples or groups of measurements.

Determination	N	m	σ	
			Original Data	Original Data Recalculated Using all Corrections
Heat capacity, cal. per deg. Cent.	28	9	0.43	0.43
Heats of solution, cal. per g.				
Dry cement	60	5	0.81	0.79
7-day paste	28	5	0.71	0.65
28-day paste	30	5	1.41	1.27
Heats of hydration, cal. per g.				
7-day	28	5	1.08	0.82
28-day	30	5	1.41	1.19

SUGGESTED CHANGES IN THE FEDERAL SPECIFICATION

Experience obtained during this investigation and in the making of hundreds of routine tests using the apparatus and method described in Federal Specification SS-C-158a indicates that certain changes are desirable in order that the accuracy and precision of the method may be improved. These changes may be grouped under (1) treatment of materials; (2) apparatus; (3) procedure, and (4) calculations.

Treatment of Materials:

Preparation of Zinc Oxide.—The preparation of the zinc oxide used for the determination of the heat capacity should be specified more closely than at present. The longer the zinc oxide is heated the less readily will it dissolve. It has been found that the following procedure gives a product that dissolves rapidly and causes the calorimeter to follow a time-temperature curve closely similar to that obtained when a hydrated cement is tested. The zinc oxide is heated for 1 hr. at 900 to 950 C., cooled and ground to pass a No. 100 sieve. Immediately before the heat capacity determination, a 7.00-g. sample of zinc oxide prepared in this manner is heated for not more than 5 min. at 900 to 950 C., cooled in a desiccator, and weighed accurately for introduction into the calorimeter.

Sampling of Dry Cement.—Particular care is necessary in sampling the dry cement in order to obtain representative material when the relatively very small (3-g). calorimeter sample is taken from a much larger quantity. It is therefore suggested that the specification be reworded to emphasize the necessity of adequate mixing of the composite cement sample before the paste is prepared or the calorimeter sample taken.

Adjustment of Temperature.—In order that the temperature of the cement paste after mixing be 70 ± 5 F., as required by the present specification, it is necessary to cool the water and cement. In addition it may be necessary to experiment with each cement sample to determine to what temperature the cement and mixing water must be cooled. To avoid these complications it is suggested that the materials be adjusted to a temperature of 70 ± 3 F. before mixing and that no requirement be made as to the temperature of the paste after mixing.

Fineness of Sample.—It has been found that grinding the hardened cement paste to pass a No. 20 sieve instead of the No. 50 sieve required by paragraph F-4 p (1) of the Federal Specification does not affect the solution rate significantly. The use of the coarser sieve permits the grinding to be done more expeditiously and with less exposure of the sample. It is, therefore, suggested that the specification be amended to require the use of the No. 20 sieve.

Apparatus:

Temperature of Room.—Since rapid changes in room temperature cause serious error, the apparatus should be placed in a room not subject to rapid or large fluctuations in temperature and should be protected from drafts.

Measurement of Temperature.—To obtain temperature measurements with the full precision of which the Beckmann thermometer is capable it is necessary to apply the corrections to which the thermometer is subject. Attention should be drawn in the specification to the need for the use of these corrections.

Speed of Stirring.—Emphasis should be placed on the need for constancy of the speed of the stirrer rather than on the desirability of some particular speed, such as "approximately 400 revolutions per minute." The heat of stirring varies as the cube of the speed of the stirrer and a variation of 5 per cent in the speed may result in approximately a 15 per cent change in the heat of stirring. The heat of stirring obtained with the different stirrers in use at 400 rpm. during the investigation was between 0.3 and 1.2 cal. per min. Assuming a low value of 0.4 cal. per min. effective over a 20-min. solution period it will be seen that a difference of 5 per cent in the average speeds during the solution and rating periods would cause an error in the heat of solution of 1.2 cal. or about 0.4 cal. per g. of cement. From the heat of stirring it is apparent that 0.001 hp. is more than sufficient to operate the stirrer. It is desirable that the specification permit the use of a speed between 300 and 500 rpm., give limits within which the variation of the speed must fall, and require that the stirrer be driven by some means other than a friction drive. The use of motors other than the series-wound or "universal" type should be encouraged because of the variability in speed of the latter under light loads. These changes in the specification would permit the builder to select a standard synchronous motor and stock gears to attain a constant speed within the desired range. Simple stroboscopes are available[5] and should be used where controlled-frequency alternating current is not available.

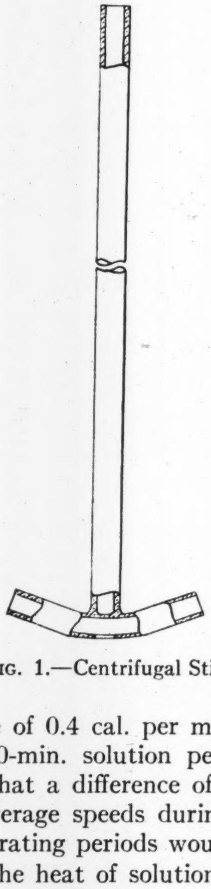

FIG. 1.—Centrifugal Stirrer.

Stirrers designed for speeds as high as 950 rpm. have been operated in this laboratory without excessive generation of heat, and others designed for speeds as low as 300 rpm. have provided sufficient agitation to give a rapid solution rate. A centrifugal stirrer, Fig. 1, has been found to be very effective in promoting a rapid solution rate at comparatively low stirring speeds (300 to 400 rpm.) since it does not permit the solid reactant to remain on the bottom of the calorimeter. Even with this stirrer, however, it is doubtful whether temperature equilibrium is attained in less than 15 min., because of low conductivity of the materials of which the calorimeter is constructed. With this more adequate agitation the time of introduction of the solid reactants may well be reduced to 1 min. or less.

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[5] The Teletype Corp., Chicago, Ill. This type is constructed by placing a shutter on the ends of a tuning fork. A small sheet of metal with a slit is placed on each of the vibrating ends of the fork. The two slits are so oriented that they coincide during each vibration of the fork. By looking through the slits at a rotating object a stroboscopic effect is produced whereby the object appears motionless, if it is rotating at the proper speed. Since in this application the constancy of the speed and not its absolute value is desired the deviation of the tuning fork from its rated frequency because of the added weight of the shutter is not important. The frequency of a tuning fork may be expected to remain constant within a few tenths of 1 per cent when subject to only normal changes in room temperature. The number of lines or marks to be placed on a rotating object, such as the disk used in this investigation, may be calculated from the formula $n = f/s$ in which n is the number of lines to be placed at uniform angular distances around the circumference of the rotating object, f is the frequency per minute with which the slits coincide, and s is the speed in rpm. at which the lines will appear stationary. Precautions, such as those suggested in footnote 3, must be observed to avoid speeds which are multiples of that desired. If the frequency of the fork, which will be reduced from its nominal value by the weight of the shutter, is not known some experimenting may be necessary with the number of lines, the speed, or even the weight of the shutter to obtain a combination by means of which the lines will appear stationary.

If the calorimeter followed the same time-temperature curve in all experiments, certain errors[6] in the measurements would compensate and not affect the value obtained for the heat of hydration. A rapid rate of solution of the zinc oxide and the dry cement tends to bring the time-temperature curves of the calorimeter during experiments using those materials into coincidence with the curves obtained with the hydrated pastes, and therefore to cause the errors to compensate. A second effect of the rapid rate of solution is to make the magnitude of the errors small so that only small or negligible errors in the heats of hydration may be caused by their lack of compensation.

Testing of Vacuum flask.—The vacuum flask should be tested from time to time. It is suggested that flasks be discarded which cool more than 0.001 C. per min. per deg. difference from room temperature after being filled with warm water, corked, and allowed to stand ½ hr. without stirring. If bulges occur in the protective coating or if the vacuum flask changes weight abnormally the acid-proof coating should be removed and renewed.

Procedure:

A stirring period of at least 15 min. should elapse in order to establish normal temperature gradients before the sample is introduced. Temperature readings at 5-min. intervals are taken after the introduction of the sample until a constant rate of change of the temperature of the calorimeter is attained. If the temperature changes occurring during three successive 5-min. intervals do not differ by more than 0.001 C. from their average expressed to the nearest 0.001 C., the rate of temperature change may be considered constant. However, determinations in which the solution period is more than 30 min. should be rejected. These requirements should be included in the specification.

Calculations:

Correction for the Temperature of the Solid Reactants.—In practice the final temperature of the calorimeter can seldom be made the same as the temperature of the solid reactant at the time it was introduced. This difficulty is recognized in section F-4r (1) of Federal Specification SS-C-158a, which contains instructions for correcting the heats of solution of dry and hydrated cements when the final temperature of the calorimeter differs from that of the solid reactant. Such corrections should be applied to all temperature differences and not be limited to those exceeding 2.0 C. The correction should also be made in calibration by multiplying the heat capacity of the 7 g. of zinc oxide (7.0 × 0.12 cal. per deg. Cent.) by the difference between the final temperature of the calorimeter and the temperature of the zinc oxide and subtracting the product from the total heat of solution of the latter, thus obtaining the amount of heat available to raise the temperature of the calorimeter. This corrected heat of reaction is then divided by the temperature rise to give the heat capacity of the calorimeter.

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[6] The errors are caused by the departure of the time-temperature curves from the ideal curve (assumed by the method of calculating the temperature rise) in which the final rate of temperature change is reached instantly. This departure has not been considered either in the Federal Specification or in the present study. However, in certain measurements it was found that the errors from this cause were almost equal in the heat capacity determination and in the measurement of the heat of solution of the dry cement. The relative errors in the heats of solution were such that values about 1 cal. per g. too high were obtained for the heat of hydration when calculated by the specification method.

In making this correction for the temperature of the hydrated paste the experimentally determined value of the heat capacity 0.4 cal. per deg. Cent. per g. of ignited cement should be used.

Correction for Temperature Coefficients of the Heat Capacity and of the Heat of Solution of Zinc Oxide.—If the isothermal heat of solution of zinc oxide at the final temperature of the calorimeter during the calibration experiment is used instead of the specification value 256.1 cal. per g., accuracy will be gained and the heat capacity of the calorimeter at the average temperature of the experiment will be obtained. This heat capacity can then be corrected by means of its temperature coefficient (+0.45 cal. per deg.[2]) to correspond with the average temperature of the heat of solution experiments.

SUMMARY

The reproducibility of measurements of the heat of hydration of portland cement made by the method described in Federal Specification SS-C-158a was investigated. The data obtained by a slightly modified method were also studied and found to be somewhat more precise.

The isothermal heat of solution of zinc oxide was determined in the concentrations of acid prescribed by the specification and found to be 256.1 cal. per g. at 30 C. with a temperature coefficient of −0.1 cal. per g. per deg. in the range 20 to 35 C.

The temperature coefficients of the heat of solution of a dry cement sample and of the hydration of portland cement were found to be −0.2 and +0.1 cal. per g. per deg., respectively, in the range 20 to 30 C.

The temperature coefficient of heat capacity of the vacuum bottle calorimeter and its contents was estimated to be +0.45 cal. per deg.[2] in the temperature range 20 to 30 C.

A number of changes in, and additions to, the Federal Specification are suggested and discussed. They are as follows:

1. The heat treatment of the zinc oxide should be specified more closely.
2. The thorough mixing of the dry cement before the calorimeter sample is taken should be emphasized.
3. The water and cement should be adjusted to 70 ± 3 F. (21 ± 1.7 C.) before mixing and no requirement should be made as to the temperature of the paste after mixing.
4. A range of stirrer speeds should be specified.
5. Methods of establishing constancy of stirrer speed should be required and limits set within which the variation in speed must fall.
6. Vacuum flasks failing to meet minimum requirements should be discarded.
7. A criterion for constancy of rate of temperature change of the calorimeter should be specified, and the permissible length of the solution period should be extended to 30 min.
8. Corrections for the heat carried into the calorimeter by the solid reactants and for the temperature coefficient of the heat of solution of the dry cement should be made in all determinations.
9. The heat of solution of zinc oxide at the final temperature of the heat capacity experiments should be used in calculating the heat capacity.
10. The hardened paste should be ground to pass the No. 20 sieve instead of the No. 50 as now required.
11. The heat of hydration should be corrected to 25 C.

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VARIATIONS IN STRENGTH OF PORTLAND CEMENTS CONFORMING TO THE SAME SPECIFICATIONS AND THE RELATION OF SUCH VARIATIONS TO CONCRETE CONTROL

By S. P. Wing[1] and Arthur Ruettgers[1]

Synopsis

This paper presents the findings from three distinct but related investigations, each involving thousands of tests.

Part I deals primarily with variations in 28-day cement strength indicated by acceptance tests of ten low-heat portland cements purchased for a large dam (dam No. 1) and with the potential effect of blending the cements. Mill strength levels ranged from 2830 to 5000 psi. The coefficient of variation in strength of 2000-bbl. lots of cement produced over a period of 1 yr. was 13.5 per cent for the average mill.

Part II deals with 28-day strength variations indicated by standard-cured 6 by 12-in. concrete control cylinders—both for dam No. 1, where the various cements were blended, and for dam No. 2, whose cement came from a single mill. Variation in cement accounted for 20 per cent of the strength variance of the control cylinders for dam No. 1, as compared with 40 per cent for dam No. 2. Seasonal temperature changes had a pronounced influence on the variations in cylinder strengths for both dams.

Part III deals with the practical significance of cement strength variations indicated by cement acceptance or concrete control tests, the supporting data being derived from long-time laboratory tests of 36 commercial portland cements in concrete under conditions simulating behavior in actual structures. It is concluded that cement strength differences revealed by cement acceptance or similar tests at early ages are of importance in the building of structures subject to curtailed curing and strength development or for which the concrete must meet an early minimum strength requirement, but that they are likely to have little bearing on the ultimate strength differences in structures such as massive dams in which the hydration is prolonged.

It is a matter of common experience that commercial portland cements of a given type from different mills, and even from the same mill, show large strength variations in routine tests made for cement acceptance or field concrete control purposes. However, it is believed that the nature, magnitude, and practical significance of these strength variations are not fully appreciated by engineers charged with the design and control of concrete construction.

The extensive construction and research activities of the U. S. Bureau of Reclamation, particularly since the building of Boulder Dam was started, have provided a vast amount of test data bearing on the subject in question. The data drawn on for this paper involve some 6000 cement acceptance strength tests applying to 5½ million bbl. of low-heat portland cement manufactured by ten mills and tested by the National Bureau of Standards for dam No. 1, an equivalent number of tests of low-heat cement supplied by a single mill for dam No. 2, about 9000 strength tests of 6 by 12-in. concrete control cylinders for the two dams, and about 3000 Denver laboratory concrete tests for comparing the strength properties of 36 commercial portland cements of three types at various test ages and under two conditions of curing (standard and mass).

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Use of Statistical Methods and Terms:

With such large numbers of tests represented by the three investigations outlined in the synopsis, any concise treatment of the subject precluded the presentation of the original test data in their entirety. Statistical procedures were accordingly employed to a considerable extent to analyze the data and extract their essence. As a consequence there is frequent use in the paper of a few statistical concepts which, while fully treated in current literature,[2] are here briefly commented on to make the main substance of the paper understandable to the average reader.

"Standard deviation,"[3] σ, is a measure of the dispersion of a group of values, and is roughly equal to 1.25 times the ordinary average deviation for a normal distribution. Conceived in another way, it represents the range (plus and minus) within which about two thirds of the values will normally depart from the average.

"Coefficient of variation,"[3] v, is simply an expression of the standard deviation as a percentage of the group average. It is a measure of relative variability.

"Variance," σ^2, is the square of the standard deviation. In this paper variance is also expressed as the square of the coefficient of variation and is used primarily to show the relative contributions of several factors to the total variation. Thus a variance of 9 (v = 3 per cent) due to experimental error, and of 16 (v = 4 per cent) due to true variation, combine to give a total variance of 25, which in turn represents a measured coefficient of variation of $(25)^{\frac{1}{2}}$ or 5 per cent.

The term "multiple correlation" refers to a statistical study whereby the combined effects of several independent variables (such as water-cement ratio and cement fineness) on a dependent variable (such as concrete strength) can be separated and the quantitative effects of each determined. The results are comparable to those which might be obtained in a testing laboratory by varying the independent factors one at a time.

Other explanatory material has been incorporated in the body of this paper as seemed to be required.

Since most of the variations dealt with in this paper are large in comparison with experimental errors, the values given are without deductions for such errors unless otherwise specifically indicated.

PART I—CEMENT STRENGTH VARIATIONS INDICATED BY CEMENT ACCEPTANCE TESTS

Object of Study:

This study was made to provide average indices of the variations in strength and related properties to be expected when portland cement is purchased from various sources under the same specifications.

Original Test Data and Summaries:

The original data come from acceptance tests made on 14,000 samples representing 5½ million bbl. of low-heat cement purchased for dam No. 1 from ten mills during a period of nearly two years. The test samples, each representing 400 bbl. of cement, were taken from conveyors feeding the storage bins at the mills. The conduct of the tests was in general accord with the procedure prescribed by Federal Specification SS-C-158 for Cements, Hydraulic; General Specifications (Methods for Sampling, Inspection, and Testing). Tests for specific surface were made on each test sample, but compression and chemical tests were made on composite samples, each representing 2000 bbl. of cement—a composite sample being obtained by combining equal amounts of cement from five successive test samples. Compression tests were on 2-in. cubes made with mortar of 0.53 water-cement ratio by weight.

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[2] A.S.T.M. Manual on Presentation of Data (April, 1943). (Issued as a separate publication.)
George W. Snedecor, "Statistical Methods," Iowa State College Press (1940).

[3] Unless otherwise indicated, the standard deviation and coefficient of variation values given in Parts I and II of the paper are values corrected for a number of observations in sample, A.S.T.M. Manual on Presentation of Data (April, 1943), p. 50.

The left half of Table I is a summary of the principal results of the cement acceptance tests. It shows the percentage of cement supplied by each mill, the average properties of the cements, the test limits imposed by the specifications, and the number of specification violations. The number of bins violating the specifications was 2.3 per cent of the total, but the departures were generally minor and no bins were rejected.

TABLE I.—AVERAGE PROPERTIES AND ESTIMATED COEFFICIENTS OF VARIATION OF LOW-HEAT CEMENTS FOR DAM NO. 1.

Mill	Proportion of Total Cement, per cent	Compressive Strength		Specific Surface, sq. cm. per g.	Computed Compound Composition, per cent				Bins Used for Analysis		Coefficient of Variation, per cent							
		28 days, psi.	Ratio of 28-day to 7-day Strength		C_2S	C_3S	C_3A	C_4AF	Number	Size, bbl.	28-day Strength (2000 bbl.) Within bin	28-day Strength (2000 bbl.) All tests	Ratio of 28-day to 7-day Strength (2000 bbl.) Within bin	Ratio of 28-day to 7-day Strength (2000 bbl.) All tests	Specific Surface (400 bbl.) Within bin	Specific Surface (400 bbl.) All tests	C_3S (2000 bbl.) Within bin	C_3S (2000 bbl.) All tests
A.....	4	2850	2.61	1990	45	26	5	18	26	7 800	6.0	17.2	3.2	7.3	3.8	7.1	3.6	7.0
B.....	22	2830	2.44	1880	46	27	5	15	41	4 000	5.9	14.3	3.2	8.4	2.5	3.4	1.9	6.0
C.....	43	3000	2.46	1920	46	30	5	13	30	4 000	6.9	11.8	4.0	8.7	2.2	4.2	3.9	5.1
D.....	7	3640	2.66	1960	45	26	6	17	18	6 000	5.4	14.3	8.1	14.2	3.9	4.6	4.3	11.8
E.....	9	3980	3.32	1990	50	27	4	14	23	2 800	5.9	13.8	7.9	14.1	3.4	5.6	5.1	7.6
F.....	6	5000	3.94	1900	51	26	5	11	6	16 000	6.1	9.4	8.4	11.6	3.4	3.8	6.5	8.1
G.....	4	3040	2.85	2020	46	27	4	17										
H.....	1	3270	2.35	1990	44	27	6	15										
I.....	2	3670	2.82	1960	46	31	5	13										
J.....	2	4690	3.81	1920	50	26	4	15										
Total and averages[a]...	100	3250	2.69	1930	47	28	5	14	..	....	6.0	13.5	5.8	10.7	3.2	4.8	4.2	7.6
Specification limits.......		2000 (min.)	1.35 (min.)	1600 (min.) (1800 for bin)	40 (min.) 65 (max.)	35 (max.)	7 (max.)	20 (max.)										
Number of bins violating specifications[b].......		5		5	2	1												

[a] Averages for columns 3 to 9 inclusive are weighted.

[b] Total number of bins violating specifications was 17, representing 2.3 per cent of all bins and 3 per cent of all cement tested. All violations on specific surface applied to bin averages.

Summarized acceptance test strengths at 28 days are also presented in Fig. 1; these are in terms of monthly means for the cement as shipped to the dam. In the bottom chart are the individual strength curves for the ten mills, and in the middle chart is a combined "cement-blend" curve derived by weighting the strengths of the individual cements in accordance with the proportionate mill

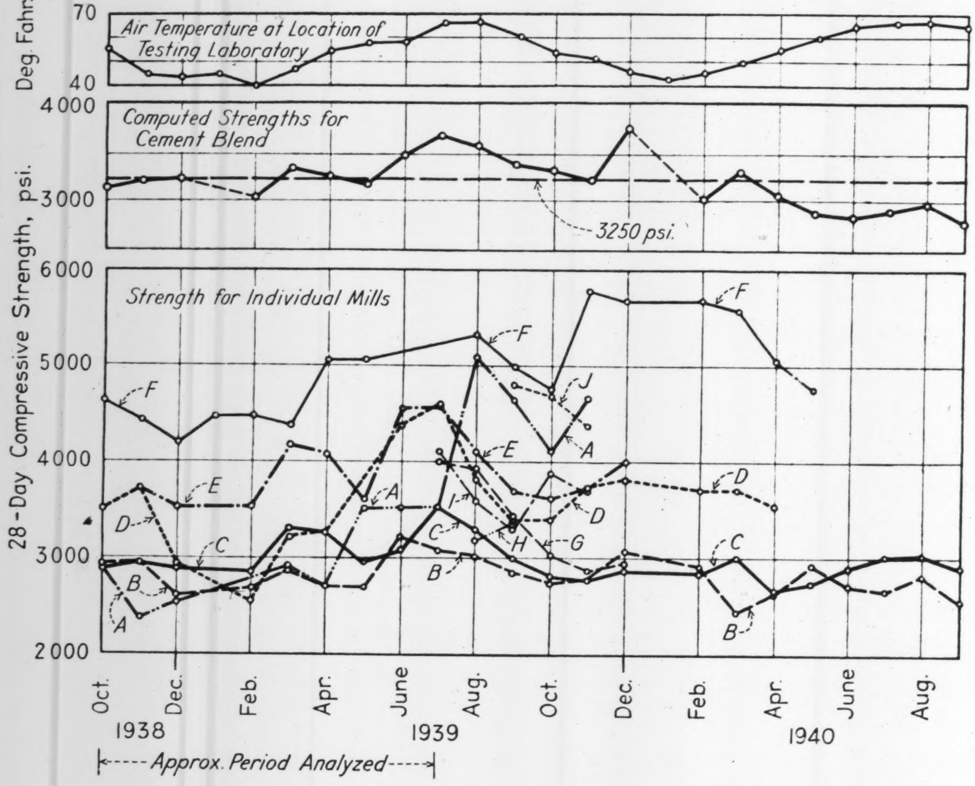


FIG. 1.—Monthly Averages of Cement Strength Acceptance Tests, Dam No. 1.

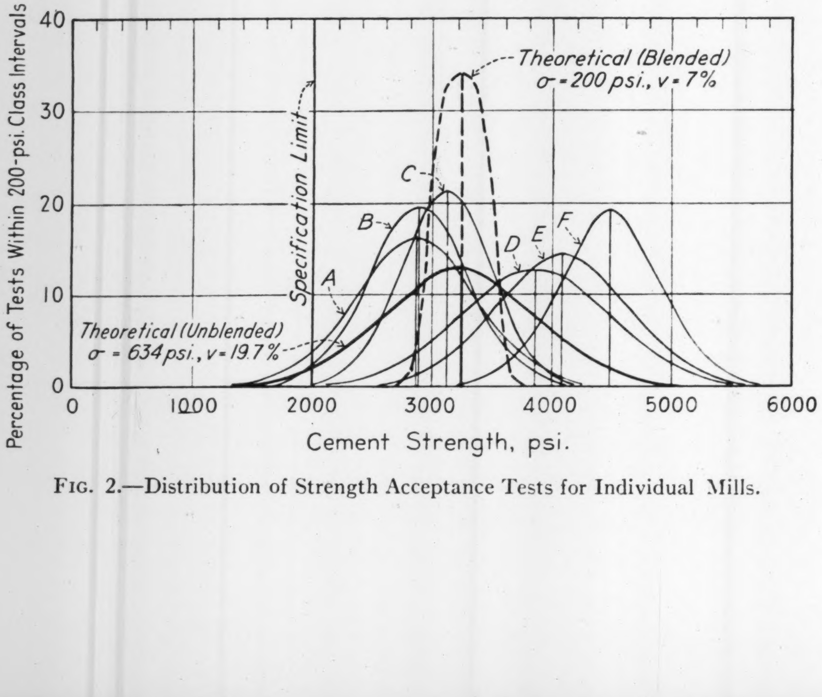


FIG. 2.—Distribution of Strength Acceptance Tests for Individual Mills.

shipments. In the top chart is a plot of the mean monthly air temperatures at the location of the testing laboratory where the physical tests for all ten cements were made.

Various features of the data presented in Table I and Fig. 1 are subsequently discussed.

Statistical Analyses of Test Data:

As seen from Fig. 1 and Table I, shipments of cement from the four mills, *G* to *J*, inclusive, extended over relatively short periods and aggregated less than 10 per cent of the total cement. The statistical analyses were therefore confined to cements from the remaining six mills, *A* to *F*, inclusive, and were further restricted to apply to a period when all these mills were shipping cement concurrently. The period selected corresponds to a period of cement manufacture of approximately one year. The quantity of cement represented is roughly 2,400,000 bbl., for which there were some 6000 test samples. About a third of the acceptance test results for the 6000 samples were utilized, these being distributed with respect to time of test and confined to bins of the same size for a given mill.

The results of analyzing variations in 28-day strength, ratio of 28-day to 7-day strength, specific surface, and C_3S content for each of the six cements are presented in the right half of Table I, in terms of coefficients of variation. An idealized conception of the 28-day strength variations of all 2000-bbl. lots of cement from the six mills, taking the mills separately and collectively, is presented in Fig. 2 in the form of frequency distribution curves, with strengths grouped in ranges of 200 psi.

Discussions of the data presented, and of the findings of various related multiple correlation studies follow.

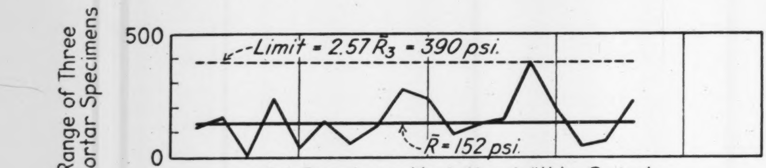
Discussion of Variations in Cement Strength:

Variation Between Mills.—Table I shows that the grand averages of the 28-day strengths for the ten cements differed greatly, ranging from 2830 to 5000 psi. The resulting coefficient of variation in mill levels is 19 per cent and the total spread is 67 per cent. The bottom chart in Fig. 1 depicts the changes in spread from month to month with shipments of cement to the job.

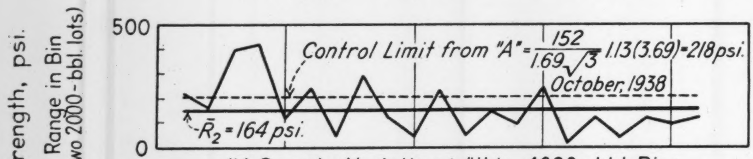
The differences between mills in average 28-day to 7-day strength ratios (Table I) is also striking, with the ratios ranging from a low of 2.35 to a high of 3.94.

Variations for Single Mills.—Coefficients of variation (Table I) were derived both for cement "within a bin," which typifies control over several days' production, and for "all tests," which applies to a year's output. The arithmetic means of these coefficients of variation for the six mills are thought to be indicative of what may be expected from the average producer of portland cement—at least for cement of the low-heat type.

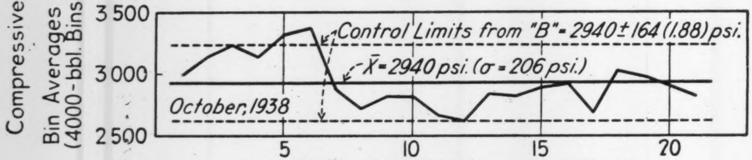
By comparing the mean coefficients of variation of 6 per cent (within bin) and 13.5 per cent (all tests), it is apparent that producers, in general, have far less control over strength variations from one period to another than during a single run. This is further exemplified by the four control charts in Fig. 3, which apply to a typical mill and were prepared in accordance with standard procedure.[2] The computations for determining, in succession, the positions of the control limits are given on the charts. In the first (top) chart the range in 28-day strength of three mortar cubes made from a single cement sample is plotted for each of 18 successive samples. The average range is 152 psi., equivalent to a coefficient of variation of 2.9 per cent for individual test specimens or $2.9/(3)^{\frac{1}{2}} = 1.7$ per cent for the averages of sets of three specimens. The latter value is the magnitude of the experimental error in testing a single cement sample. Excellent control is evidenced by all plotted points being within the control limit. The next three control charts are typical of the strength variations of samples (each representing 2000 bbl.) within a bin, bins within a month, and monthly averages during a 2-yr. period, respectively. The fact that the control limits on all three charts were exceeded indicates that in each case new causes of variation of one kind or another were introduced, the general trend toward higher strengths in summer and lower strengths in winter shown by the bottom chart being worthy of note. Combining the apparent variations within a day, from day to day within a month, and from month to month within a year, to roughly approximate the expected variation in strength of 2000-bbl. lots of cement from a year's average strength gives a coefficient of variation of $(5^2 + 7^2 + 6.6^2)^{\frac{1}{2}}$ or 11 per cent.



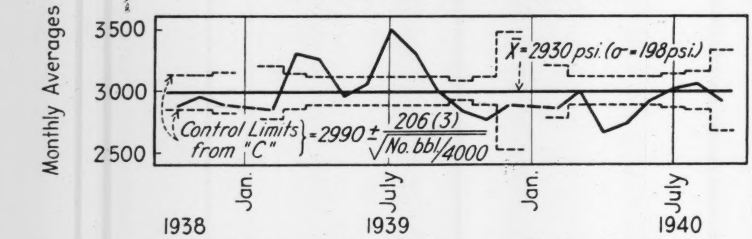
(a) Specimen Variation Within Sample



(b) Sample Variation Within 4000-bbl. Bins



(c) Bin to Bin Variation Within Month



(d) Variation of Monthly Averages During 2-yr. Period

FIG. 3.—Cement Strength Control Charts.

With respect to the ratio of 28-day to 7-day strength, Table I shows a mean coefficient of variation of 10.7 per cent for "all tests." By considering this value as a reasonable measure of the bin-to-bin variation for the average mill, it follows that for one bin out of three the strength ratio could be expected to fall outside the values 2.69 ± 10.7 per cent, or outside the ratios 2.4 to 3.0. Thus, forecasts of 28-day cement strengths from 7-day test values are subject to errors of these magnitudes.

The 28-day strength distribution curves for mills A to F in Fig. 2 were derived by computation from average mill strengths in conjunction with the coefficients of variation listed in Table I for "all tests." A sample plot of the actual distribution for one mill did not differ significantly from the theoretical curve. The curves are effective in depicting the working ranges of the mills with respect to strength and their relation to the minimum strength requirement of the specifications. To illustrate, there was no likelihood of mill F failing to meet the limit of 2000 psi., whereas mill A assumed the risk of having about 6 per cent of the tests (involving three 200-psi. class intervals) show failure.

Combined Variations (Between and Within Mills).—The heavy solid-line curve in Fig. 2 with an indicated coefficient of variation of 19.7 per cent[4] represents the theoretical strength distribution of all 2000-bbl. lots of cement supplied by the six mills as a whole. As such, the curve takes into account the proportionate mill deliveries. It shows, for example, that for an average strength of 3250 psi., 2 per cent of all 2000-bbl. lots produced might be expected to fall within the 1900 to 2100-psi. strength range and about the same amount in the 4400 to 4600 psi. range. Actually, the reported test results showed somewhat fewer low and more high values.

The potential effect of blending the products of the six mills, assuming each mill's output to be in statistical control, is approximated by the heavy dashed curve in Fig. 2 which shows a coefficient of variation of only 7 per cent, as compared with the 19.7 per cent for unblended cement. The curve represents the computed distribution of the average strengths of sets of six tests chosen at random from all the tests.

Figure 1 shows that as few as two and as many as ten mills made concurrent shipments of cement to the dam and that the individual mill strength levels changed appreciably from month to month, both of these factors having their effect on the strength curve for the cement blend. The subject of cement blending is more fully discussed in Part II of this paper.

Causes of Variation in Cement Strength:

Variation in Specific Surface.—For individual mills, variation in specific surface was found generally to be the principal cause of fluctuation in strength. By referring to the coefficients of variation listed under "all tests" in Table I it will be noted that mills A and E, which showed the largest variations in specific surface and about average variations in C_3S, also showed large variations in strength.

Multiple correlation studies for mill A cement, using specific surface and C_3S as the independent variables, showed that a unit change of 100 sq. cm. per g. in specific surface caused a change of 219 psi. in 28-day strength and accounted for a third of the total strength variance.

Variation in C_3S.—Variation in C_3S was the important cause of variation in strength of mill D cement, a multiple correlation with C_3S and specific surface as the independent variables showing that about two thirds of the strength variance (square of the multiple correlation 0.80) was due to this cause. For a unit increase in C_3S (and corresponding decrease in C_2S), the increase in 28-day strength was 137 psi. A similar correlation for mill A cement gave a value of 108 psi.

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[4] Strength data on 6 by 12-in. concrete cylinders at 0.60 water-cement ratio by P. H. Bates (Report on "Cooperative Tests of Thirty-Two Portland Cements by Forty-Seven Laboratories," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part I, p. 270 (1928)) yield coefficients of variation between cements of 19.6, 19.8, 13.4, and 8.4 per cent at 3, 7, 28, and 365 days, respectively.

Cyclic Phenomena.—The striking similarity in strength trends for eight of the ten cements from August to October of 1939 (see Fig. 1) led to the discovery that there was general correspondence between the cement strength – time curves and the outdoor temperature curve (top graph) for the place where the physical tests of the cements were made. The average strength drop of the eight cements during the period mentioned amounts to about 500 psi. or 14 per cent. In addition, cements A, D, and E were found to give statistically significant strength correlations with temperature for their entire records, the average correlation being 0.65. These apparent relationships are closer than can be expected by chance, yet no adequate reason for them has been found, an investigation having indicated that the testing laboratory conditions and technique were well within existing standards. Possibly weather variations affect some features of mill control.

All that can be stated at present is that cyclic phenomena (see also Fig. 3(*d*)) seem to have affected the cement strengths reported and that the effects are large in relation to the usual errors in cement testing.

Variation in Conditions of Cement Manufacture.—It is apparent from a comparison of the average strength, specific surface, and computed compound composition values listed in Table I, particularly those for cements A and D, E and F, and B and J, that the reported differences in composition are decidedly inadequate as a means of explaining the large differences in mill strength levels. Perhaps some enlightened reader will supply the answer to the riddle. At the moment the authors are obliged to dismiss the subject summarily by attributing the discrepancies largely to differences in raw materials, cement manufacturing procedures, and clinker and cement storage.

PART II—STRENGTH VARIATIONS INDICATED BY FIELD CONCRETE CONTROL CYLINDERS

Object of Study:

The principal object of this study was to investigate the extent to which differences in cement were responsible for variations in the 28-day strength of field concrete control cylinders.

Scope of Study:

Isolation of the cement factor necessitated an analysis of the principal factors contributing to the variations in concrete strength.

In addition to data for dam No. 1, data for a second dam, dam No. 2, are presented to afford a direct comparison of strength variations under two different conditions of cement supply, the cement for dam No. 2 having been supplied by a single mill.

General Conditions Governing Concrete Manufacture:

Concrete Production Facilities.—The mass concrete to which the strength test data apply was produced in large automatically-operated mixing plants equipped with the most advanced facilities for proportioning the ingredients by weight, for ribbon-feeding the batched materials, and for controlling the consistency of the concrete. All mixers were of 4-yd. capacity, and the normal period of mixing was from 2 to 2½ min.

Concrete Control Procedure.—While the procedure for controlling concrete manufacture included the routine testing of 6 by 12-in. concrete cylinders at several ages, only the 28-day test data were used for this paper. The cylinders were made from samples of mixer discharge, after wet-screening the concrete to remove aggregate larger than $1\frac{1}{2}$ in., and except for a brief period of preliminary storage at the mixing plants, were cured in fog rooms at standard temperature.

TABLE II.—SUMMARY OF 28-DAY CONCRETE CONTROL CYLINDER STRENGTHS AND RELATED DATA—DAMS NOS. 1 AND 2.

	Dam No. 1	Dam No. 2
Concrete strength:		
Number of 6 by 12-in. test cylinders	4600	4000
Average strength, psi.	4150	3160
Minimum and maximum, psi.	2530–6150	1680–5160
Coefficient of variation of all tests, per cent	12.5	18.2
Variance (v^2) of all tests	156	330
Coefficient of variation of monthly means, per cent	7.1	15.2
Standard deviation of monthly means, psi.	293	481
Cement Strength:		
Average, psi.	3250	2980
Coefficient of variation of 2000-bbl. lots, per cent	10 (Estimate for blend)	14
Coefficient of variation of monthly means, per cent	7.9	10.9
Standard deviation of monthly means, psi.	253	324
Water-cement ratio of concrete (by weight):		
Average	0.537	0.593
Coefficient of variation of monthly means, per cent	2.8	3.3
Standard deviation of monthly means	0.015	0.019
Concrete temperature at mixer:		
Average, deg. Fahr.	64.0	63.6
Minimum and maximum monthly, deg. Fahr.	55–72	52–78
Coefficient of variaton of monthly means, per cent	6.2	11.9
Standard deviation of monthly means, deg. Fahr.	4.0	7.6
Specific surface of cement:		
Average, sq. cm. per g.	1930	1950
Coefficient of variation of monthly means, per cent	1.6	5.1
Standard deviation of monthly means, sq. cm. per g.	29	99
Concrete slump at mixer:		
Average, in.	2.05	2.00
Coefficient of variation of monthly means, per cent	4.4	7.5
Standard deviation of monthly means, in.	0.089	0.15

Concrete strength-prediction equations:
Dam No. 1.—Concrete$_{28}$ = 10,790 − 14,600 water-cement ratio + 0.38 Cement$_{28}$
Dam No. 2.—Concrete$_{28}$ = 7200 − 11,200 water-cement ratio + 0.87 Cement$_{28}$
Statistics applying to prediction equations:
Dam No. 1.—Correlation (22 months) = 0.70 ± 0.11
Standard error of estimate = 210 psi. (5.1 per cent)
Dam No. 2.—Correlation (25 months) = 0.92 ± 0.03
Standard error of estimate = 194 psi. (6.1per cent)

The general procedure governing mix proportions was to maintain the weight ratio of cement to total aggregate constant, and to regulate the consistency to that required for concrete of fixed slump at points of placement. Thus, while the cement content of the concrete (approximately 1 bbl. per cu. yd.) and related factors, such as heat generation, were nearly constant, the water-cement ratio fluctuated with changes in temperature, cement fineness, etc., as later discussed.

Cement Blending.—The cement blending procedure conformed to that at Boulder Dam, where experience with the simultaneous use of cements from various mills in lining the diversion tunnels revealed striking differences in water requirement, workability, bleeding, and rate of stiffening of the fresh concrete, as well as differences in color, heat evolution, strength development, and cracking of the concrete after set had taken place.

Cement as received at dam No. 1 was unloaded into eight silos, each of 5000-bbl. capacity, the different brands of cement being kept separate in so far as the storage facilities permitted. Cement was released from these silos in proportion to the total amounts supplied by the various mills and fed directly or by branch conveyors to a common screw conveyor for blending, after which the product was delivered to two finish-storage silos.

Presentation of Data:

Figures 1 and 2, giving strength data on the cements for dam No. 1, were presented and discussed in Part I of this paper.

Table II is a compilation of summarized test data and statistical data on the 28-day concrete cylinder strengths for dams Nos. 1 and 2, and on the five principal factors related thereto; namely, water-cement ratio, slump, temperature of the concrete at the mixing plants, and strength and specific surface of the cement.

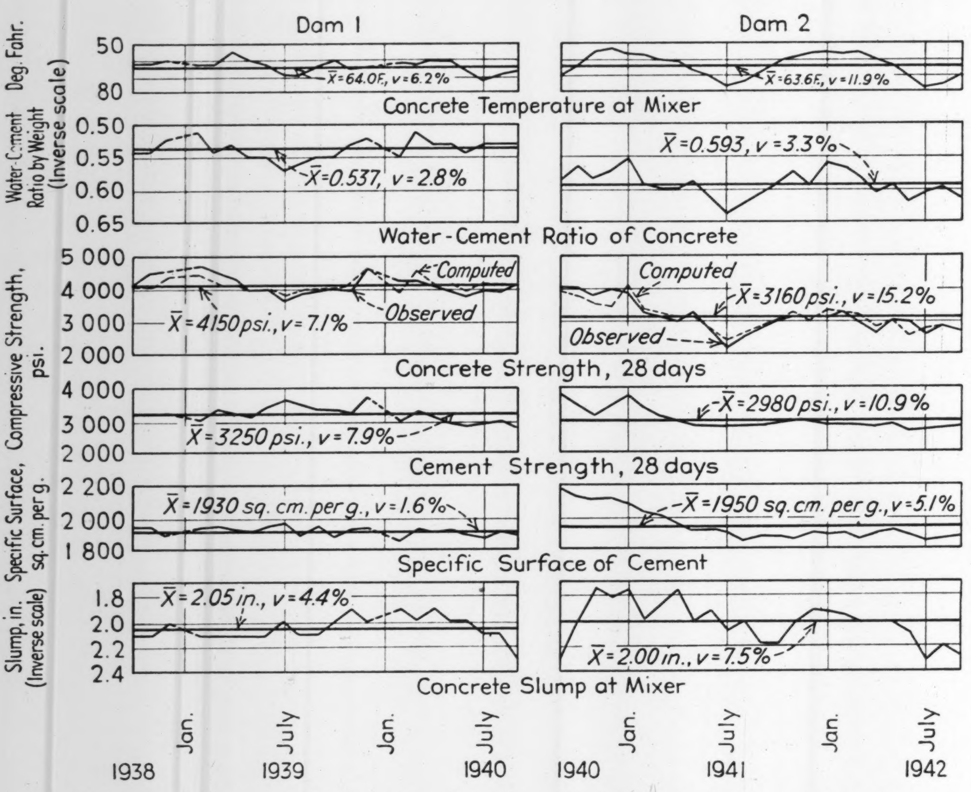


FIG. 4.—Comparison of 28-day Strengths of Concrete Control Cylinders and Related Factors for Dams Nos. 1 and 2.

In Fig. 4 the test data for dams Nos. 1 and 2 are presented graphically, in terms of mean monthly values throughout the selected periods of construction. The reader should note that the concrete temperature, water-cement ratio, and slump graphs were drawn with the scales inverted to facilitate comparison of the trends in the six graphs.

The ensuing discussions are based largely on the above-mentioned data.

Causes of Variation in Concrete Strength:

General.—Before discussing specific causes of variation in concrete strength, the three control charts in Fig. 5 are presented to illustrate the type of concrete control found possible at dam No. 1 over a 2-yr. period. The upper chart, showing within-batch control, was prepared by plotting for 34 batches of concrete sampled during a single month, the range in strength of two cylinders made from each batch. The mean range of 184 psi. corresponds to a coefficient of variation of 3.1 per cent. The fact that all plotted points came within the computed control limit[2] and that the testing error was of the order of 3 to 4 per cent is evidence that mixing and testing procedures were well controlled.

Control limits for the middle and bottom charts were set from the data of each preceding chart in a manner such that if new causes of variation were introduced, points could be expected to fall outside the limits. That new causes were introduced from batch to batch and from month to month, of a magnitude represented by a coefficient of variation of about 7 per cent in each case, is apparent.

Variation in Concrete Temperature at Mixer.—Variation in temperature of the concrete at the mixing plants (closely associated with variation in mean air temperature) apparently affected the concrete strength directly as well as indirectly (through water-cement ratio), with the indirect effect predominating and both effects acting in the same direction.

Perhaps the most prominent and interesting feature of Fig. 4 is the general similarity of the curves for concrete temperature, concrete strength, water-cement ratio, and slump. The cyclic change in concrete strength, with low strength in summer and high strength in winter, is of course also present in the bottom chart of Fig. 5.

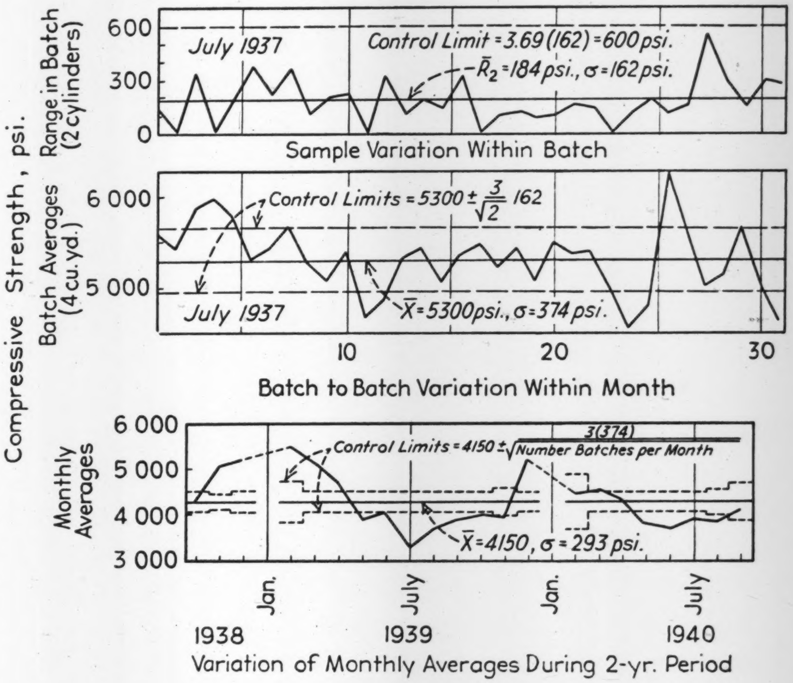


Fig. 5.—Concrete-Strength Control Charts.

The apparent cyclic range in strength for dam No. 1 is 600 psi. with a range in monthly average concrete temperature of 17 F., the corresponding values for dam No. 2 being 900 psi. and 26 F. For both dams the unit change is about 35 psi. per deg.

The indirect effect of change in temperature is two-fold. With rising temperature, for example, the water-cement ratio was increased both by reason of the increase in water requirement of the mix for concrete of the same slump at the mixer, and of the requirement for a greater difference in slump between the mixer and the points of concrete placement to compensate for greater losses of slump in transit.

A plot of water-cement ratios against concrete temperature for dam No. 1 showed an increase in water-cement ratio of 0.017 for each increase in temperature of 10 F. For dam No. 2 the value was 0.020.

Variation in Specific Surface of Cement.—Specific surface has two indirect effects on concrete strength—through cement strength and through the water-cement ratio. The combined effect may be substantial, as it was at dam No. 2.

The similarity of the curves for cement strength and specific surface in Fig. 4 is especially marked for dam No. 2.

A multiple correlation of water-cement ratio as a function of specific surface and slump for dam No. 2 showed a decrease of 0.007 in water-cement ratio for each increase of 100 sq. cm. per g. in specific surface. Expressed in terms of concrete strength by the use of other derived data (later presented), this decrease of 0.007 in water-cement ratio per 100 sq. cm. per g. increase in specific surface corresponds to a strength increase of 80 psi.

With the aid of still further studies, the combined unit contribution of specific surface to concrete strength (operating through cement strength and water-cement ratio) for dam No. 2 was found to be 265 psi. per 100 sq. cm. per g., or 8.4 per cent of the average 28-day control cylinder strength.

Variation in Concrete Slump.—Since the cement content was virtually constant and the slump at the mixer varied within relatively narrow limits, the effect of changes in slump on concrete strength was essentially that resulting from induced changes in the water-cement ratio. No quantitative data on the effect of change in slump are presented, as correlation studies did not yield significant values.

The range in slump at the mixing plant from winter to summer, based on monthly averages, was 0.2 in. for dam No. 1 and 0.4 in. for dam No. 2. The figures are indicative of the seasonal difference in slump losses.

Variations in Water-Cement Ratio and Cement Strength.—Of the five causes of variation considered (exclusive of experimental error), water-cement ratio and cement strength are the two whose effects on concrete strength are regarded as being wholly direct. The nature of the studies for analyzing the contributions of these two factors makes it convenient to treat them together.

The results of a multiple correlation, for each dam, of concrete strength as a function of water-cement ratio and cement strength, using monthly averages, are given at the bottom of Table II in the form of strength-prediction equations and related statistics. These equations were used to compute the monthly concrete strengths, for comparison with the actual values, and the results are shown by the dashed lines in the graphs for concrete strength in Fig. 4. That variations in water-cement ratio and cement strength together account for a substantial part of the observed month to month variation in cylinder strengths is indicated by the general agreement of the curves. The tendency for the computed strengths to be too low for the winter months and too high for the summer months lends support to the belief that concrete temperature had a direct effect on cylinder strength, the indirect effects of concrete temperature, specific surface, and slump having been provided for in the choice of cement strength and water-cement ratio as the independent variables.

Referring to the strength prediction equations, the figures 14,600 for dam No. 1 and 11,200 for dam No. 2 by which the water-cement ratio is multiplied indicates that the strength of the concrete was reduced by 146 and 112 psi., respectively, for each 0.01 increase in water-cement ratio. These values represent the same percentage decrease for each job, amounting to 3.5 per cent for each 0.01 increase in water-cement ratio.

Similarly the multipliers 0.38 and 0.87 in the prediction equations indicate that at constant water-cement ratio each 100 psi. variation in strength of the cement reflected as 38 and 87 psi. variation in strength of the concrete made from it. The first coefficient (0.38) is much less reliable than the second, as shown by the relative values of the corresponding correlation coefficients. It is believed that the data justify the conclusion that from 0.8 to 0.9 of the variation in 28-day cement strength found by standard tests on 2-in. mortar cubes will appear as variation in the 28-day strength of 6 by 12-in. concrete cylinders of 3000 to 4000 psi. average strength.

Relative Contributions to Variation in Concrete Strength:

Analysis of Variance.—Table III gives the results of an analysis made to determine the relative importance of the principal factors causing variation in concrete cylinder strengths at dams Nos. 1 and 2. Although the statistical analysis was not complete, the values given represent the authors' best estimate of variance distribution within the average month and between the monthly means.

The total variance to be accounted for is the square of the coefficient of variation in strength of individual cylinders for the 2-yr. period examined. At dam No. 1 (see Table II), the strengths varied from 2530 psi. to 6150 psi., a range of 87 per cent; and the coefficient of variation was 12.5 per cent, giving a variance of 156. At dam No. 2 the variation was from 1680 psi. to 5160 psi., a range of 110 per cent; the coefficient of variation was 18.2 per cent, and the variance 330.

Table III shows that whereas the variances within the average month were very similar for the two dams, both as to total magnitude (104 and 98) and as to distribution (cement 25 and 23; water-cement ratio 40 and 30), the month-to-month variances were greatly different. At dam No. 1 the month-to-month variance totaled 52, or one third the total to be accounted for; at dam No. 2 it was over four times as great, 232, and about two thirds of the total.

TABLE III.—APPROXIMATE DISTRIBUTION OF VARIANCE OF 28-DAY STRENGTH OF 6 BY 12-IN. CONCRETE CYLINDERS.

Factors Causing Variation	Dam No. 1				Dam No. 2			
	Within month	Month to month	Total	Per Cent	Within month	Month to month	Total	Per Cent
Cement[a]	25	6	31	20	23	108	131	40
Water-cement ratio[b]	40	13	53	33	30	37	67	20
Concrete temperature	4	8	12	8	10	49	59	18
Mixing and testing (experimental error)	20	..	20	13	20	..	20	6
Unaccounted for	15	25	40	26	15	38	53	16
Total	104	52	156	100	98	232	330	100

NOTE.—Square root of variance gives approximate coefficient of variation.

[a] Variance values include effect of variation in water-cement ratio due to cement.

[b] Variance values exclude effect of variation in water-cement ratio due to cement.

The variances given for cement include not only the direct effect on concrete strength of variations in cement strength but also the indirect effect of variations in specific surface on concrete strength through the effect on water-cement ratio. On this basis, the cement contributed 20 per cent of the total variance in cylinder strengths for dam No. 1 as compared with 40 per cent for dam No. 2, the corresponding contributions of water-cement ratio being 33 and 20. Thus the water-cement factor predominated at dam No. 1 and the cement factor at dam No. 2.

Effect of Cement Supply and Cement Blending.—The large difference in month-to-month variances contributed by the cement for dams Nos. 1 and 2 (6 against 108, Table III) is evidence of substantial differences in cement supply and cement blending. It cannot be laid to difference in mill control, as the month-to-month variations in cement strength and specific surface for dam No. 2 cement were comparable with those for cement supplied by the average mill for dam No. 1.

The theoretical value of the coefficient of variation in strength of 2000-bbl. lots of cement from six mills for dam No. 1 was 19.7 per cent (Fig. 2) without blending and 7 per cent with blending. With an average of 15 blends per month, the expected coefficient of variation of monthly mean strengths would be $7/(15)^{1/2}$ or about 2 per cent. The actual value was 3.9 per cent for the 9-month period when the six mills alone were shipping (see Fig. 1) and 7.9 per cent for the entire 2-yr. period, during which time from two to ten mills shipped concurrently.

At dam No. 2, on the other hand, the coefficient of variation in strength of all 2000-bbl. lots of cement was 14 per cent, and for the monthly means was 10.9 per cent. The latter figure is in contrast with a theoretical value of about 1½ per cent, assuming a blending procedure equivalent to that assumed for dam No. 1. The coefficient of variation in specific surface, based on monthly means, was also substantially higher for dam No. 2 cement, being 5.1 per cent as compared with 1.6 per cent for dam No. 1 cement.

Thus though lack of statistical control in cement manufacture, variation in shipping conditions, and unavoidable imperfections in cement blending prevented full realization of the theoretical benefits of blending cements from several sources, the net effect was still decidedly in favor of dam No. 1. Had no blending facilities been provided at dam No. 1, it is estimated that the coefficient of variation in control cylinder strengths would have been about 20 per cent instead of 12.5 per cent, or higher than for dam No. 2.

Variance Reduction Possibilities.—The variance study indicates that there is no royal road to the production of concrete which over a period of a year or more would be much more uniform than that at dam No. 1. Even it if had been possible to remove all variance ascribed to the cement, the coefficient of variation in cylinder strengths, 12.5 per cent, would have been reduced to only $(156-31)^{1/2}$, or 11.2 per cent. And if in addition the variances due to water-cement ratio and concrete temperature had been eliminated, the coefficient of variation remaining would still have been 7.7 per cent.

In Part III of this paper mention is made of conditions under which variations in concrete control cylinder strengths due to cement variations may be of large importance. Assuming for the moment that this were true of the conditions prevailing at dam No. 2, and that it had been desirable to compensate for major causes of variation, the indications are that a material reduction in cylinder strength variation could have been realized by adjusting the water-cement ratio and mix proportions periodically to offset the effects of variations in cement fineness and seasonal temperature. Such a procedure, of course, ignores the possibility of securing increased uniformity in portland cements through improvements in manufacture and specification requirements.

Part III—Practical Significance of Cement Strength Variations

Parts I and II have disclosed the nature, magnitude, and causes of variations in strength of cements purchased for two dams and the extent to which such variations were reflected in the strength of 28-day standard-cured concrete cylinders. However, the quantitative data given are not directly applicable to the actual mass-cured concrete in the dams, as will be clear from what follows.

Had the concrete cylinders been subject to a minimum 28-day strength requirement, or had the concrete been used, let us say, in the structural frame of a building under conditions such that the degree of hydration ultimately attained was equivalent to 28 days' standard curing, then obviously the data presented would have been directly applicable. In other words, the practical significance of cement variations indicated by cement acceptance tests or tests of standard-cured concrete cylinders is dependent on the character of the structure and its environment, particularly with respect to conditions affecting hydration of the cement, and on the nature of any concrete control strength stipulations imposed.

Object and Scope of Study:

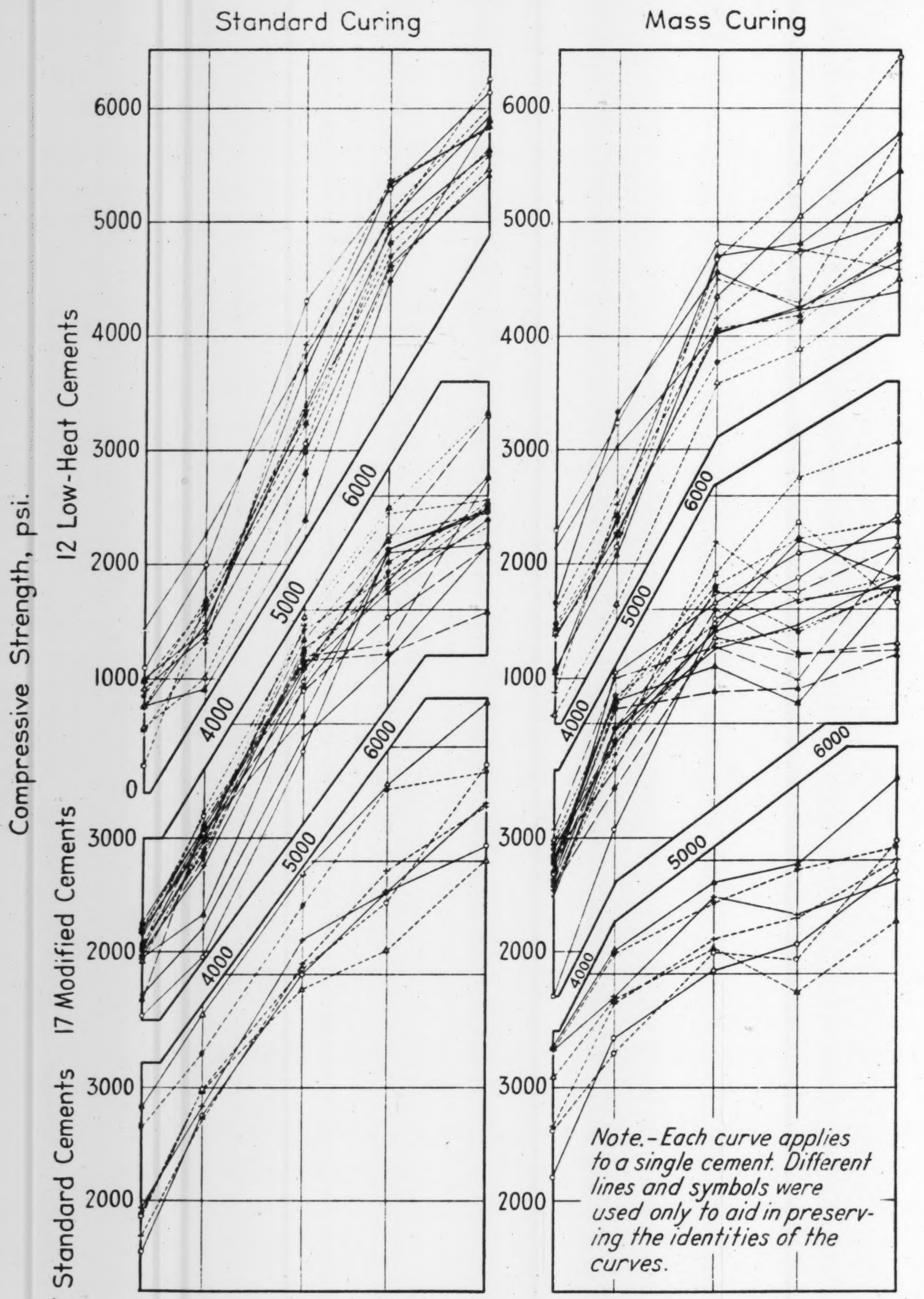
To assist in evaluating the effect of cement strength variations indicated by cement acceptance or concrete control tests in terms of strength variations of the concrete in actual structures, an analysis was made of the age-strength relations shown by laboratory tests conducted over a 10-yr. period for comparing the strength properties of 36 commercial portland cements in concrete under both standard-curing and mass-curing conditions. The analysis was aimed not only to show the change in coefficient of variation in strength due to cement with continued strength development of the concrete, but also to throw some light on the extent to which one cement or group of cements might be expected to maintain an initial strength advantage over another.

TABLE IV.—RANGE IN SPECIFIC SURFACE AND COMPUTED COMPOUND COMPOSITION OF CEMENTS, AND IN WATER-CEMENT RATIO OF CONCRETE.

Item	Twelve Low-Heat Cements	Seven Standard Cements	Seventeen Modified Cements	All Thirty-six Cements
Specific surface, sq. cm. per g.:				
Max.	2150	1980	2170	2170
Min.	1860	1530	1750	1530
Avg.	1990	1795	1975	1945
Compound composition, per cent:				
C_3S				
Max.	30	45	51	51
Min.	10	29	29	10
Avg.	20	39	43	35
C_2S				
Max.	61	43	46	61
Min.	42	28	21	21
Avg.	52	33	30	38
C_3A				
Max.	8	14	9	14
Min.	3	10	3	3
Avg.	5	11	6	7
C_4AF				
Max.	18	9	18	18
Min.	6	7	10	6
Avg.	15	8	14	13
Water-cement ratio by weight:				
Max.	0.57	0.60	0.56	0.60
Min.	0.52	0.52	0.50	0.50
Avg.	0.549	0.549	0.537	0.543

Details of Laboratory Tests and Strength Results:

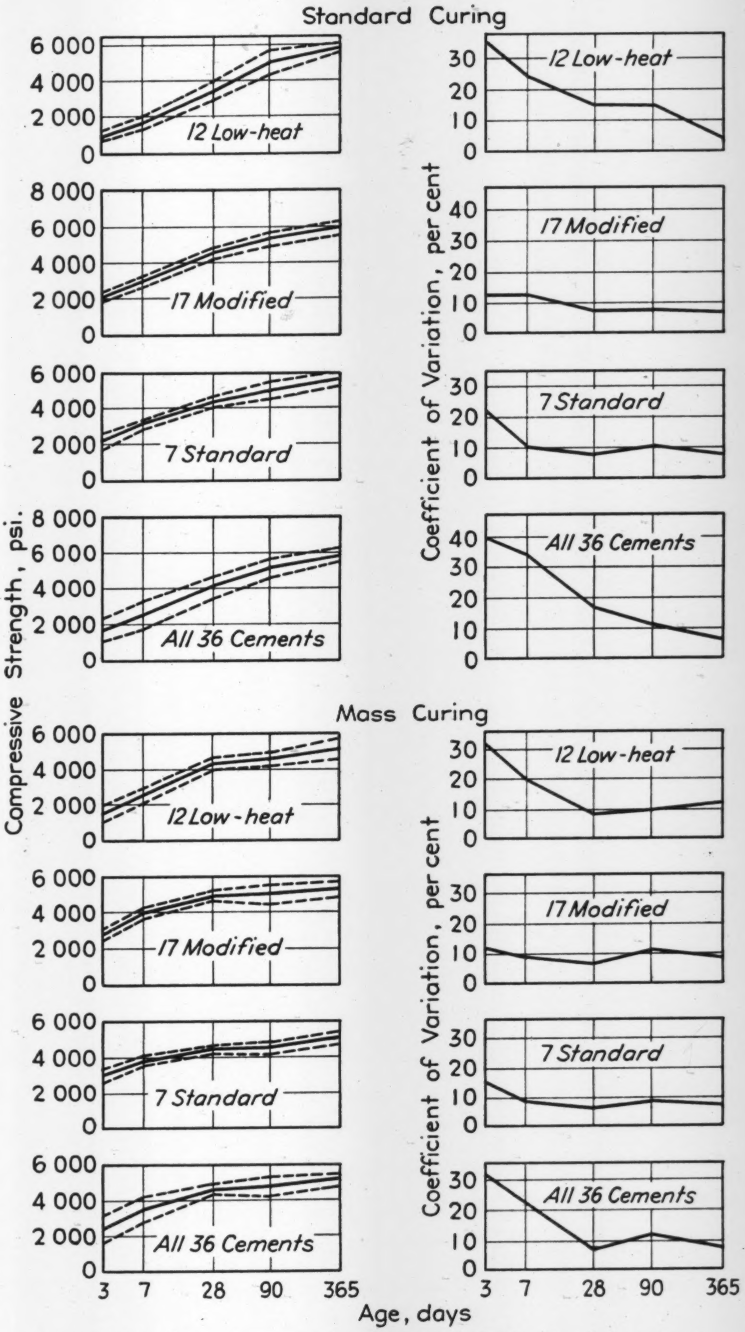
Cement.—Of the 36 cements used, 15 were manufactured in California, 5 in Washington, 2 in Texas, and 10 in eastern states, the remaining 4 being blends of cements from Washington, Oregon, and Colorado. Three general types of cement are represented, there being 12 low-heat, 7 standard, and 17 modified cements. The ranges in cement fineness (specific surface by Wagner



Note.-Each curve applies to a single cement. Different lines and symbols were used only to aid in preserving the identities of the curves.

FIG. 6.—Denver Cement Tests. Strength-age relations for individual cements.

Each plotted point represents the average strength of nine test specimens with the exception of the 3-day strength for which there were six specimens.



Note:-Solid lines show average strength. Ordinate distances between solid and broken lines represent standard deviations.

FIG. 7.—Denver Cement Tests—Analysis of Cement Strength Variations.

turbidimeter) and computed compound composition are shown in Table IV.

Aggregate.—The aggregates consisted of natural sand and gravel, separated into the various standard sizes and recombined by weight for each batch of concrete. Test specimens for the last eleven cements tested were made with aggregates from a second source.

Number and Size of Test Specimens.—With minor exceptions, as later indicated, there were nine test specimens—three each of 2 by 4-in., 3 by 6-in., and 6 by 12-in. cylinders—for each test age and type of curing in testing a single cement. For the 3-day tests only, the 6 by 12-in. cylinders were omitted; and for the last eleven cements tested, the 2 by 4-in. cylinders were replaced with 2-in. cubes. The total number of specimens was 3024.

Mix Proportions.—Aggregates were graded to maximum sizes of $\frac{3}{16}$ in., $\frac{3}{4}$ in., and $1\frac{1}{2}$ in. to accommodate the three sizes of test specimens. The water-cement ratio was established for each cement by preliminary test as that required to yield a fixed slump in mass concrete containing 1 bbl. of cement per cubic yard and a combined aggregate of fixed grading. The predetermined water-cement ratio for a given cement was used for all concretes made with that cement, and the mix proportions for a given maximum size of aggregate were chosen to produce concretes having approximately the same consistency. Table IV shows the range in water-cement ratios for cements of each type and for all 36 cements.

Curing.—Test specimens were cured continuously until tested in compression. Standard curing was in fog rooms. Mass-cured specimens, cast in sheet-metal containers, were sealed to prevent moisture loss or gain. These specimens were exposed to a rising and subsequent lowering temperature cycle which started and ended at 70 F., the cycle terminating at the time of test, or at 28 days for specimens tested at later ages.

Strength Results.—The strength test results (adjusted for eleven of the cements to correct for the effect of change in aggregate supply) are shown for each of the 36 cements by the curves in Fig. 6, each plotted point representing the average strength of nine test specimens, with the exception of the 3-day strength for which there were six specimens. Different lines and symbols were used for the curves merely to aid in preserving the identities of the curves, hence they carry no significance other than that a given cement is represented by the same combination of line type and symbol in the curves for standard curing and mass curing.

Analysis of Test Results:

Strength Variations Between Individual Cements at Ages of Three Days to One Year.—The solid-line curves in the left-hand diagrams of Fig. 7 were derived by averaging the strengths for cements of each type and for all 36 cements. The broken-line curves, drawn above and below the solid-line curves by amounts equal to the computed standard deviations in strength at the several test ages, represent the strength boundaries within which about two thirds of the strength curves for the individual cements theoretically would fall. It will be observed from the spacing of the curves that the standard deviations within a cement group are generally fairly uniform, which means that if the strength variations were expressed as percentages of the rising average strengths, the resulting values (coefficients of variation) would decrease with age. This is borne out by the curves in the right-hand diagrams, which show pronounced drops in coefficient of variation in most cases, the points of least variation being reached at 1 yr. for standard curing and at 28 days for mass curing. Under standard curing, considering the 36 cements as a whole, the coefficient dropped from 40 per cent at 3 days to 16 per cent at 28 days, and finally to 6 per cent at 1 yr. Similarly, under mass curing the drop was from 33 per cent at 3 days to 7 per cent at 28 days and to 8 per cent at 1 yr. The 12 low-heat cements showed reductions of about the same order as the entire 36 cements, while the 17 modified and 7 standard cements showed only small or no reductions beyond 28 days under standard curing or beyond 7 days under mass curing.

Change in Relative Strength Positions of Cements with Continued Hydration.—Figures 8 and 9 disclose certain pertinent trends not readily or fully discernible from Figs. 6 and 7.

Figure 8 provides a direct comparison of the average strength curves for the three types of cement. There are four features of special interest:

1. The gradual reduction in strength spread from values of 1200 to 1500 psi. at 3 days to a value of 300 psi. at 1 yr., due primarily to the ability of the low-heat cement to overcome its initial strength handicap.

2. The gradual change in the relative position of standard cement from top place at 3 days to bottom place at 1 yr.

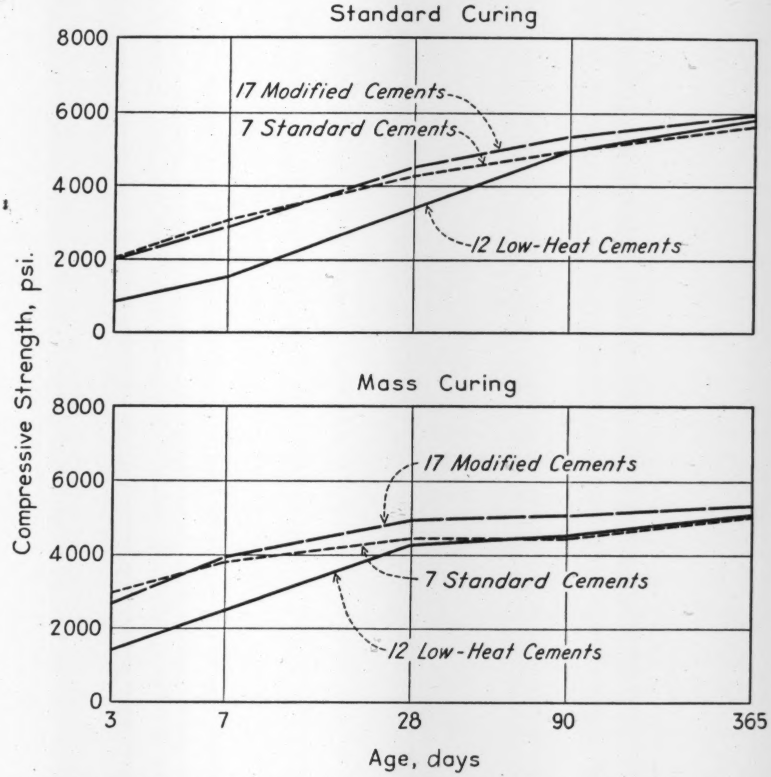


FIG. 8.—Denver Cement Tests. Average age-strength relations for three cement types.

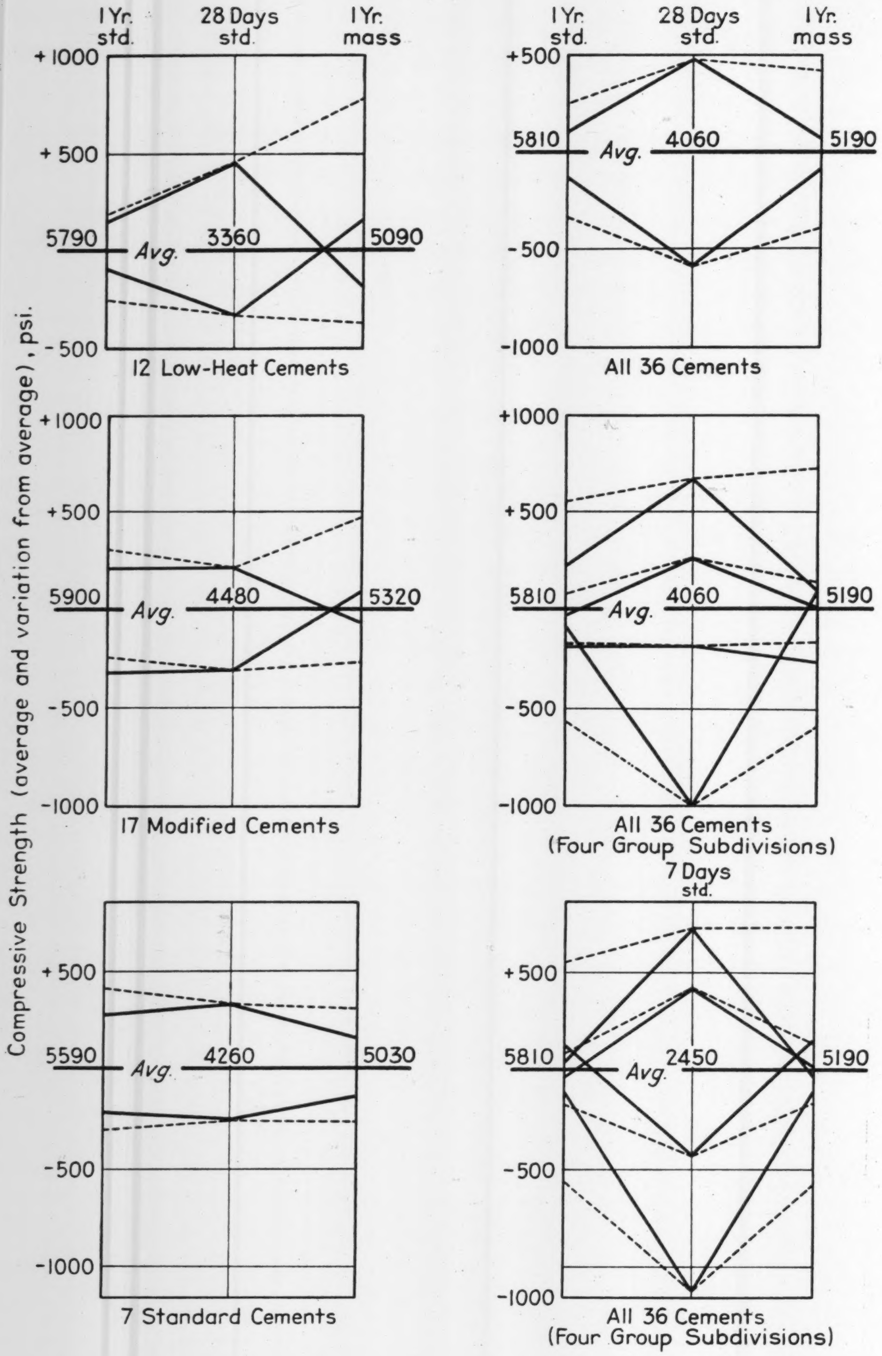


FIG. 9.—General Changes in Cement-Strength Positions from Early to Late Ages.

3. The effect of mass curing in accelerating strength development at early ages but causing a sacrifice in strength at later ages, as compared with standard curing.

4. The similarity of change in cement strength positions for standard curing and mass curing, the principal difference being one of time. (Compare 3-days mass curing with 7-days standard curing, 7-days mass curing with 28-days standard curing, etc.)

As cement strength data obtainable from cement acceptance and concrete control tests are generally based on standard-cured specimens broken at early ages (3 to 28 days) and as the data in Parts I and II of this paper relate to 28-day strengths, the changes in relative strength positions of cements in the Denver test series were studied by comparing the strength positions at 28 days (one study using 7 days) under standard curing with those attained at 1 yr. under both standard and mass curing. Comparisons were made for the cements of each type considered separately, as well as for the 36 cements as a whole. The results of the studies are shown in condensed form by the diagrams in Fig. 9.

Derivation of the diagrams requires explanation. The method used in connection with the three diagrams at the left of the figure and the top diagram at the right was somewhat different from that applying to the two lower diagrams at the right and will be explained first.

Referring to the top left diagram for the purpose of illustration, the three numerical values shown just above the heavy horizontal line are, from left to right, the average strengths of the 12 low-heat cements for 1 yr. standard curing, 28 days standard curing, and 1 yr. mass curing, respectively. The dash lines connect points of average strength of all cements whose strengths were either above or below the general averages for the three conditions of test, while each of the four solid sloping lines connects the point of average strength of those cements whose strengths were above (or below) the general average at 28 days with the point of average strength of the very same cements at 1 yr. The extent of inward departure of any solid sloping line from its corresponding dash line, therefore, reflects the extent to which the individual cements failed to maintain their relative strength positions with continued curing.

Construction of the two lower diagrams at the right was the same in principle, the only difference being that the subdivision of cement strengths was carried a step further. To illustrate, the uppermost plotted point in the vertical center line of a diagram represents the average strength of those cements whose strengths were higher than that of the average of all cements showing strengths above the grand average.

It is noteworthy that except for three cases out of thirty-two the trend is toward equalization of cement strength positions at a late age, irrespective of differences in cement composition, specific surface, or water requirement. Some of the comparisons even show a tendency toward ultimate reversal of the 28-day strength position, especially where mass curing is involved. It should be emphasized that the trends are only general trends and that some individual cements do not conform, as is evident from a critical examination of the curves in Fig. 6.

One thing is certain: that is, that relative cement strength values obtained from cement acceptance or similar tests at early ages are in themselves no reliable criterion of relative strength standings in concrete at late ages with continued hydration.

Actual versus Estimated Variations in Strength of Concrete Cores:

As some concrete cores were drilled from various locations in dam No. 1 and both low-heat and modified cements[5] were represented, it is pertinent to inquire whether the variations in core strength were in reasonable agreement with values to be expected from the findings of the present investigations.

By referring to Fig. 7 and comparing the coefficients of variation in strength at 28-days standard curing with those from 28-days to 1-yr. mass curing, it will be noted that for low-heat cements the coefficient was reduced about one third, for modified cements it remained about the same, and for all cements combined it was reduced about one half.

As previously stated, the coefficient of variation found for the 28-day concrete control cylinders at dam No. 1, using low-heat cement, was 12.5 per cent. The corresponding coefficient for the period when modified cement[5] was used is not available but is estimated at 12 per cent. From these two coefficients together with known values of the average 28-day strengths and total numbers of control cylinders for the low-heat and modified cement concretes, it is roughly estimated that the over-all coefficient of variation (for all concrete control cylinders made at dam No. 1) was 20 per cent.

From the foregoing data, the coefficients of variation in strength to be expected from large numbers of cores, for any age from 28 days to 1 yr., were roughly estimated. The predicted values are 8 per cent for low-heat cement concrete, 12 per cent for modified cement concrete, and 10 per cent for all concrete in the dam. While the actual core data are limited, the results are reassuring.

One set of six 22-in. diameter cores, 735 to 797 days old, drilled from modified-cement concrete in three different blocks in the dam, gave a coefficient of variation in compressive strength of 4.7 per cent. A second set of six cores of the same size, 623 days old, taken from a single block where low-heat cement was used, gave a coefficient of 3.1 per cent. The coefficient for all twelve cores without making adjustment for difference in age was 6.0 per cent.

The coefficients of variation and related data for three sets of 6 by 12-in. drill cores taken from sections of the dam where only low-heat cement was used are as follows:

	Age 105 to 141 days	Age 270 to 292 days	Age 628 days
Total number of cores	37	6	15
Number of drill holes represented	6	3	5
Number of blocks represented	1	3	1
Coefficient of variation, per cent	10.8	14.2	10.0

The much higher coefficients of variation for the 6-in. than the 22-in. diameter cores are ascribed in part to the 6-in diameter being too small for comparative strength tests of mass concrete containing 6-in. cobbles.

CONCLUSIONS

The investigation of strength variations shown by: (1) acceptance tests of ten low-heat cements purchased concurrently under the same specifications, (2) field tests of 6 by 12-in. concrete control cylinders made at two dams, and (3) comparative laboratory tests of 36 commercial cements in concrete, leads to the following conclusions:

1. The average 28-day strength of ten cements supplied to meet a minimum strength requirement of 2000 psi. was

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[5] Modified cement, not treated in Parts I and II of this paper, was used for dam No. 1 prior to the use of low-heat cement. Core strength data applying to both types of cements are introduced to illustrate a more extended application of the Denver test findings (Fig. 7).

3250 psi. Strength levels of the mills ranged from 2830 to 5000 psi., the coefficient of variation between them being 19 per cent. The average mill supplied cement with a coefficient of variation in strength of 13.5 per cent, as measured by tests on composite samples representing 2000 bbl. of cement. It was impossible reliably to predict the range of strength for a mill in any month from tests made in a prior month, as the strengths were not in statistical control.

2. The coefficient of variation in 28-day strength for all 2000 bbl. lots of cement from all mills was about 20 per cent. Blending of the cements prior to use at dam No. 1 was estimated to have reduced the coefficient of variation to about 10 per cent.

3. High variations in 28-day strength of cement from individual mills were found to be associated with high variations in specific surface and C_3S content, these two factors accounting for 65 to 80 per cent of the total variance for the two mills investigated. The average specific surface of ten cements supplied to meet a minimum requirement of 1600 sq. cm. per g. for a single sample and 1800 for a single bin was 1930 sq. cm. per g. For the average mill the coefficient of variation in specific surface for all test samples was 4.8 per cent. Multiple correlation with 28-day strength and C_3S content for one cement showed a strength increase of 219 psi. for each 100 sq. cm. per g. increase in specific surface, or 1.5 per cent strength change for each 1 per cent change in specific surface. The average C_3S content for the ten cements (to meet a maximum requirement of 0.35) was 0.28, and the coefficient of variation in C_3S for all tests for the average mill was 7.6 per cent. Multiple correlation with 28-day strength and specific surface for two cements showed about 125 psi. increase in strength for each 0.01 increase in C_3S content.

4. The ratios of 28-day to 7-day strengths for the ten mills ranged from 2.35 to 3.94, and averaged 2.69. The ratio was not constant even for a single mill.

5. Multiple correlations, for two dams, of 28-day concrete control cylinder strengths with field water-cement ratios and 28-day cement strengths showed high significance. With respect to dam No. 1, for which the cement was a blend of the products of two to ten mills, the coefficient of variation for all concrete cylinders during a 2-yr. period was 12.5 per cent; 20 per cent of the variance in cylinder strength was attributed to cement variations; and the estimated water-cement ratio variance was nearly twice the estimated cement variance. With respect to dam No. 2, for which the cement was furnished by a single supplier, the coefficient of variation for the concrete cylinders was 18.2 per cent; 40 per cent of the variance in cylinder strength was ascribed to cement variations; the estimated cement variance was twice the estimated water-cement ratio variance; and the correlation study showed an increase of 87 psi. in concrete strength for each 100 psi. increase in cement strength.

6. The apparent effect of increase in concrete temperature at the mixer from winter to summer, combined with the effect of using additional water to provide concrete of constant slump at the point of placement without changing the cement content, was to decrease the 28-day strength of control cylinders at dam No. 1 about 600 psi., the corresponding range in monthly average temperature being 17 F. At dam No. 2, the decrease in strength was about 900 psi. with a temperature range of 26 F.

7. Comparative laboratory tests of 36 commercial cements in concrete continuously cured and designed to apply to field concrete of fixed slump and cement

content showed marked reduction in coefficient of variation in cement strength with age, both for standard and mass curing (see Fig. 7). The reduction was greatest for low-heat cements, least for modified cements, and intermediate for standard cements. Under standard curing the coefficients of váriation at 28 days were generally less than half the values at 3 days but as much as three times the value at 1 yr. At 28 days to 1 yr. under mass curing, the coefficients were generally comparable to those at 1 yr. under standard curing. A study of the relative strength positions of the individual cements at 28 days, standard curing, with those at 1 yr. for both standard and mass curing, showed that the initial strength positions were not maintained. It also indicated that there was a general trend, both for cements within each type and between types, toward strength equalization at late ages or, in the case of mass curing, toward ultimate reversal in strength position.

8. The variations in strength of concrete cores drilled from dam No. 1 were much less than those for corresponding 28-day concrete control cylinders, and were about in line with values anticipated from the comparative laboratory cement studies.

9. The practical significance of cement variations indicated by cement acceptance tests or tests of standard-cured concrete cylinders is dependent on the character of the structure and its environment, the nature of any concrete control strength stipulations imposed, and the procedure for mix proportioning. In the construction of a concrete building frame, with limited opportunity for continued strength development, or of any structure built under a 7-day or 28-day minimum concrete strength requirement, cement variations should be reflected to a high degree in the strength of the concrete in the building or the control cylinders, as the case may be, unless compensated for by appropriate changes in water-cement ratio. On the other hand, cement variations indicated by the usual tests at early ages are likely to give an exaggerated and erroneous picture of the ultimate strength differences of the concrete in a massive dam, or a highway pavement in a humid climate, when the cement content and slump are held constant.

Acknowledgments:

The authors are indebted to Valens Jones, Robert E. Kennedy, A. H. Yeates, and O. H. Millikan for substantial assistance rendered in the preparation of material used in this paper. Cement acceptance tests were made by the National Bureau of Standards for the U. S. Bureau of Reclamation. The tests of the 36 cements in concrete, made in the Bureau laboratories at Denver, were directed by H. S. Meissner under the supervision of R. F. Blanks and Arthur Ruettgers. All engineering research and design are under the direction of J. L. Savage, Chief Designing Engineer. S. O. Harper is Chief Engineer of the Bureau of Reclamation, and H. W. Bashore is Commissioner of Reclamation.

DISCUSSION

Mr. W. C. Hanna.[1]—There is one thing that occurs to me that probably accounts for some of the variations mentioned. The tests made with the turbidimeter may not represent the true fineness. Some other instrument, such as the Blaine machine[2] could show a difference of as much as 400 sq. cm. per g. of cement between two samples which have about the same specific surface as measured by the instrument used by the authors in making their fineness determinations.

Mr. W. M. Kinney.[3]—I should like to raise this thought. If I am correctly informed, most of the tests here reported were made on low-heat cement. Is it not a fact that most of this cement was made at mills which were just starting to make low-heat cement?

Up until a few years ago most cement companies had had little experience in the manufacture of low-heat cement. We all know that when there is a change from one product to another in any manufacturing process there is a period of inexperience which may lead to greater than normal variation in the resulting product. If it were found that in the tests on earlier samples greater variations were noted than on the later ones, that might be of some significance.

Chairman P. H. Bates.[4]—I might emphasize what has been stated by the authors in regard to the strength-level variations between the various mills. One mill that employed an old-fashioned warehouse type of storage was the mill that consistently had very much higher strength than the other mills. There is nothing in any calculation from composition, no matter how it is calculated, that would give that high strength. That was particularly true of the 28-day strength which was extremely high for a portland cement and yet it was a low-heat cement.

However, coming back to one point raised by Mr. Kinney, I would say again in looking over the results presented that they are as expected. I do not think that the spread is large, I think if you took any seven mills, over 2 yr. making standard portland cement, you would find a greater variation. One reason for this is that these mills were making a special cement and the staff was most careful at all times. But when they return to making standard cement, the staff relaxes in its supervision and the plant operates with much less exacting control.

Mr. John Tucker, Jr.[5] *(by letter)*.—Messrs. Wing and Ruettgers have done a worthy service in the presentation and analysis of such a mass of important data representing a first step in the attempt to apply the principle of statistical control to concrete fabrication. The paper shows what has been done in a systematic attempt to establish uniform concrete strength on large projects, and from the paper it is clearly demonstrable that new methods of testing the cement are necessary if the concrete strength and other properties are to be predicted with reasonable certainty. It also shows the way to needed research.

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[1] Chief Chemist and Chemical Engineer, California Portland Cement Co., Colton, Calif.

[2] Raymond L. Blaine, "A Simplified Air Permeability Fineness Apparatus," ASTM Bulletin, No. 123, August, 1943, p. 51.

[3] Vice-President and General Manager, Portland Cement Assn., Chicago, Ill.

[4] Chief, Clay and Silicate Products Div., National Bureau of Standards, Washington, D. C.

[5] Chief, Cement and Concreting Materials Section, National Bureau of Standards, Washington, D. C.

The Wagner turbidimeter is inadequate to measure the specific surface of the finer particles, and cannot be used to predict the water requirements of a cement as influenced by the fineness. It is a well-known fact that a cement ground by closed circuit and one ground in an open circuit, both of the same Wagner specific surface, will differ appreciably in their water requirements and that to develop equal strengths in lots from one batch of clinker ground in open and in closed circuits, the specific surfaces may differ by several hundred square centimeters per gram. This may have been the explanation as to why the specific surface reported by Wing and Ruettgers fails to give an adequate indication of strength differences. The Blaine or Lea-Nurse fineness apparatus suggest themselves as improved alternate methods.

The computed compound compositions, one of the sets of variables presented by the authors, were computed without regard to the glass content of the cement. As Ward[6] has demonstrated, this method may cause the computed amounts of the several compounds to differ largely from the much more significant ones computed with recognition of the glass phase.

If more complete data had been obtained on the grading and compound composition of the cement, closer correlation between cement properties and concrete strength probably would have been found.

The statistical treatment in the paper, particularly in Figs. 3, 4, and 5, leaves something to be desired. The authors have not explained, nor given the references to, the numerous factors entering into their formulas for control limits, and it is hoped that they will do this in their closing discussion. The constants in the figures are without meaning unless reference be had to the 1943 A.S.T.M. Manual on Presentation of Data.[7]

The authors consider the control in Fig. 3 (*a*) as excellent. In view of the fact that one of the 18 observations lies upon the control line (a probability of $\frac{1}{18}$, or 5.6 per cent) and that the control line represents the equivalent of a range of $3\,\sigma$, a probability of 0.997, the control is not to be classed as "excellent."

In the text concerning Fig. 3(*a*) the authors state that ". . . . the coefficient of variation . . . . is the magnitude of the experimental error." Obviously it is only a measure of the dispersion of the results of tests of single samples of cement.

In Fig. 3 (*a*), and in the middle portion of Fig. 5, the control limits are computed by two methods: from constant A_2 of Table I, and from Eq. 1, p. 51 of Supplement B of the Manual,[7] respectively. The results will be the same by both methods and the use of both may be confusing.

With reference to the use of ranges in the paper, it may be noted that Tippett warns against the use of ranges: "It is very clear that the method of ranges is markedly inferior to the method of moments, and, sample for sample, to the method of ranking." The Manual also classes the use of ranges second to that of moments. Particularly, when specimens are grouped in pairs, the method given by Deming and Birge[8] is to be preferred. This is practically identical with classical method, using the equation $\sigma_i = \sqrt{\frac{\Sigma\delta^2}{n-1}} : \frac{\sqrt{2}}{2}$ where σ_i is the standard deviation of individuals and δ is the difference between the individuals in a pair, and n is the number of pairs.

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[6] George W. Ward, "Effect of Heat Treatment and Cooling Rate on the Microscopic Structure of Portland Cement Clinker," *Journal of Research*, Nat. Bureau of Standards, Vol. 26, No. 1, January, 1941, p. 49. (R.P. 1358.)

[7] A.S.T.M. Manual on Presentation of Data (1943). (Issued as separate publication.)

[8] Deming and Birge, "The Statistical Theory of Errors," *Review of Modern Physics*, Vol. 6, No. 3, July, 1934.

The "theoretical (blended)" frequency curve of Fig. 2 may be misleading. It is true that the curve is "theoretical" and correct as to theory, but the question arises from which hypothesis does the theory originate. If groups of six tests of cement from any *one* mill were averaged, then the spread in these strengths would be decreased by the factor $\frac{1}{\sqrt{6}}$ and decreased in proportion, as the "theoretical (unblended)" curve of Fig. 2 had been contracted to the width of the "theoretical (blended)" curve of the same figure. Of interest to the investigator would be the *actual* spread in strength of tests made of cement blended in a manner identical to that by which it was blended in actual concreting operations. It is then likely that the spread of strengths would be as wide as the spread for the samples from each of the individual mills.

The authors, in comparing the strength dispersions of 6-in. and 22-in. diameter cores, do not take cognizance of a theory for the difference in their dispersions suggested by two investigators.[9,10] This theory, which is supported by experimental data, postulates that the standard deviation of the strength of a specimen of n square inches is equal to the standard deviation of that of unit area, divided by the square root of n. Thus if the 6-in. diameter cores have a coefficient of variation v then the 22-in. specimens will have a standard deviation of $v \cdot \frac{6}{22}$ or $0.273\ v$.

Now if, instead of considering the two groups of 22-in. diameter cylinders made from two types of cements as one group, we find the mean coefficient of variation by the more logical method of adding the variances and taking the square root, $\left(\sqrt{\frac{(4.7)^2 + (3.1)^2}{2}}\right)$ we obtain a coefficient of variation of 4.0 per cent. Similarly we get a coefficient of variation of 11.0 per cent for the three groups of 6-in. cores.

Computing the theoretical coefficient of variation for the 22-in. diameter specimens from the 11.0 per cent value of the 6-in. diameter specimens, we get 11.0 × 0.273 = 3.0 per cent, not far from the 4.0 per cent value just obtained; particularly when we consider the reliability of the values expressed by their standard deviations:

For 6-in. cylinders $v = 11.0 \pm 1.0$
For 22-in. cylinders $v = 6.0 \pm 1.1$
(calculated from one group of 12)
For 22-in. cylinders $v = 4.0 \pm 0.8$
(calculated from two groups of 6)
For 22-in. cylinders (theoretical)
$v = 3.0 \pm 0.28$
(calculated from 6-in. specimens)

The analysis in the paper is an excellent illustration of the great practical value of the latest issue of the A.S.T.M. Manual on the Presentation of Data.

MR. H. W. LEAVITT[11] (*by letter*).—Seldom, if ever, has it been possible to assemble related masses of concrete and portland cement test data in the magnitude found in this paper by Messrs. Wing and Ruettgers. They should be especially complimented on the statistical analysis of this great quantity of test data. Instead of presenting voluminous tables of test results, they have given us only the statistical facts and relationships. These figures—standard deviations, coefficients of variation, and multiple correlation equations—constitute the

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[9] John Tucker, Jr., *Journal*, Franklin Inst., Vol. 204, December, 1927, p. 751.

[10] W. Weibul, *Proceedings*, Royal Swedish Institute for Engineering Research, No. 151 (1939).

[11] Secretary, Maine Technology Experiment Station, University of Maine, Orono, Me.

extracted essence and value of this great mass of data. Future investigators will use these basic coefficients for purposes of comparison. Undoubtedly some of these results will eventually be helpful in writing the specification limits for acceptance of materials, or for bases of rejection of faulty material.

The concrete strength prediction equations given at the foot of Table II should be of great value and interest to users of concrete in general. With the use of an equation of this type, 28-day strengths may be predicted from two test values—namely, water-cement ratio and 28-day cement strength. Formerly prediction equations involved 7-day strength results, as for example Slater's equation:[12]

$$f'_c = f + 30\sqrt{f}$$

and the Maine equation[13]

$$S_{28} = 0.846\,(S_7) + 1517$$

A comparison might be made concerning the variation to be found in the strength tests on portland cement from any one plant. Maine Technology Experiment Station *Paper No. 18*[14] gives yearly coefficients of variation on standard tensile strength tests on one brand of regular portland cement produced from one plant for a period of 8 yr. These values of v range from 4.1 to 8.0 and show greater concordance than those given in Table I in Wing and Ruettgers' paper. Another comparison of data might be made using the compressive strengths of field concrete control cylinders reported in Maine Technology Experiment Station *Bulletin No. 33.*[13] The coefficient of variation then obtained on 400 field concrete cylinders from one project was found to be 11.3, whereas in Table II, Wing and Ruettgers report a corresponding figure of 12.5 per cent for dam No. 1, and 18.2 per cent for dam No. 2.

These two comparisons are presented for the purpose of showing how future analysts and investigators will compare their data with the basic findings of this most interesting and valuable paper.

MR. N. T. STADTFELD[15] (*by letter*).—The authors have done a workmanlike job in compiling data which cover a large number of tests. In spite of the vast amount of work involved, the report leaves one without fundamental increase in knowledge. The authors themselves have realized this in calling for help to explain the extreme differences in mill strength levels.

It is believed that the great handicap under which nearly all investigators have to work lies in the fact that it is assumed that specifications as now written for portland cement limit the product sufficiently to allow deductions as to its behavior in the field from its behavior in the laboratory. Such is by no means the case and increased knowledge can only be obtained by limiting the many variables which enter into production. True research in this field starts with complete cooperation between producer and consumer. If the matter is left entirely in the hands of the producer, progress appears to be held back by a desire to protect the fellow manufacturer with a difficult quarry, antiquated equipment and poor quality product—witness the fact that Richard K. Meade's treatise on "Portland Cement, Its Composition, Raw Materials, Manufacture, Testing and Analysis," is still the standard work although written in 1906 and in spite of the many improvements made since then. The consumer working alone, on a finished product, is helpless in conducting meaningful research.

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[12] W. A. Slater, "Relation of 7-Day to 28-Day Compressive Strength of Mortar and Concrete," *Proceedings*, Am. Concrete Inst., Vol. 22, p. 437 (1926).

[13] H. Walter Leavitt, "Predictive Values and Variability of Field Concrete Cylinders from Maine Gravel Aggregates," Maine Technology Experiment Station, *Bulletin No. 33*, May, 1937.

[14] H. Walter Leavitt, "A Statistical Study of Tension Tests of One Brand of Portland Cement," Maine Technology Experiment Station, *Paper No. 18*, May, 1936.

[15] Division Engineer, Inspection Div., Board of Water Supply, City of New York, N. Y.

It is not evident from the report that when unusual variations were found in the product, which could not be explained by variation in test procedure, any attempt was made to obtain from the manufacturer information as to the probable cause. It would have been preferable to include mill control in the specifications. In 1937 the late Mr. Thaddeus Merriman, unwilling "to dismiss the subject summarily by attributing the discrepancies largely to differences in raw materials, cement manufacturing procedures, and clinker and cement storage" as do the authors, began where they leave off and attempted to eliminate, or at least account for, these discrepancies when he drew up the cement specifications for the Board of Water Supply of the City of New York. There has been much opposition from many quarters to specifying mill control but it is a fact that only by insisting on regularity of kiln temperatures and kiln rotations are some of the variables eliminated. Combine with this the stipulation that the finished product pass the Merriman sugar test, which for Type II cement is a definite indication as to the efficiency of burning, and it becomes evident that a product is obtained which is much less likely to be underburned and variable. In addition, the care demanded for Board of Water Supply clinker is such that the loss on ignition never exceeds the allowed maximum of 0.9 per cent.

While over 6,000,000 bbl. of cement made under Board of Water Supply specification have shown a remarkable regularity in chemical composition and a total absence of laitance when used in concrete in the field, it does not mean that there is no strength variation between the products of ten mills which have supplied the cements. There will always be a normal cement for each mill, which in some cases may contain as little as 21.6 per cent of silica and in others approach 24 per cent of silica. In addition each plant has its own peculiarities and a surface area of 1560 sq. cm. per g. for one mill where open circuit is used in grinding may give a product comparable in strength to that of another mill with air-separators where the surface area is 1850 sq. cm. per g.

The fluctuations in the product of a mill, when noticed in the laboratory, should immediately be taken up with the manufacturer and knowledge gained thereby. For instance, our laboratory found that cement from a certain plant would go along in normal fashion up to a silica content of 23.8 per cent. At this point an optimum condition seemed to prevail and as soon as the silica reached 24.0 per cent a sudden reduction in early strength would occur. Care in blending raw materials prevented future trouble. Again, overloading of the finishing mills with subsequent elimination of spread in particle size, as sometimes occurred with air-separators, resulted in a harsh product evident in the laboratory while making neat cement test briquets. Such a cement would show excessive bleeding in concrete and increased fineness was the remedy in this case.

Another instance comes to mind where a very troublesome condition of premature set was overcome by lowering the grinding temperature of the clinker and decreasing simultaneously the SO_3 content of the cement.

The above cases are simply quoted as examples of the true value of a consumers control laboratory. They do not fill many pages, but may well be of more value than a record of thousands of routine tests made on the basis of an inadequate specification.

Inasmuch as the authors have requested comments, the above is submitted as a possible approach. In addition it is my belief that, although the blending of various cements on the work may reduce the variations in concrete strength, from the point of view of tracing back failures in a structure such blending is an abomination.

MR. P. J. FREEMAN[16] (*by letter*).—The authors of the paper are to be congratulated on their tenacity and tirelessness in studying and analyzing the vast amount of data which they had available from the tests made on such large quantities of cement. We attempted to do the same thing some years ago and analyzed our tests on moderate-heat and low-heat cement. We assembled a considerable amount of information from the tests made up to that time which represented several million barrels of cement, but as our construction program broadened and the number of barrels of cement being tested and used increased, our enthusiasm for the problem waned and we are still holding in our files copies of test results on cement representing about thirteen million barrels. It seems probable that these test results could be made available to anyone who had the time and resources to analyze them.

When a large hydraulic structure is made from cement produced at different mills, it is difficult and sometimes impossible to identify the tests made on the concrete directly with the cement tests. But in the case of one large dam we were able to do this due to the nature of the methods used in handling the cement and placing the concrete. A careful analysis and study was made and the results of the compression tests on the cement mortar cubes were correlated with the concrete strengths produced from the same cement. The 7- and 28-day mortar strengths of 2-in. cubes made with graded Ottawa sand mortar and a plastic mix paralleled to a very satisfactory degree the strengths of 6 by 12-in. concrete cylinders molded in the field. The 7-day mortar tests show a range in compressive strength from 1600 to 3200 psi., while the 7-day concrete strengths range from 2100 to 3300 psi. The 28-day mortar and concrete tests show a similar close relationship which indicates that it is possible to predict the earlier concrete strengths from the cement tests when 2-in. cubes made with graded Ottawa sand mortar and a plastic mix are used. The same general relationship between mortar strengths and concrete strengths appeared to exist at later ages but on account of the fewer number of concrete tests no definite statement can be made.

All cement was bin-tested and none shipped which did not fully comply with the specifications and in turn the concrete was tested from samples and cores taken from the field. An additional quantity of data has been accumulated on temperature studies of concrete structures. As the construction program enlarged, the methods for handling and using cement at the job-site increased the difficulties encountered in correlating the test results on cement with the concrete tests, and unless some of our people develop the same energy possessed by the authors of the paper it seems doubtful whether these data will be made public.

MR. DUFF A. ABRAMS[17] (*by letter*).—The writer had occasion four years ago to refer to some of the topics covered by this paper in his discussion of "Mixer Efficiency or Mortar-Mix Tests," (*Journal*, American Concrete Institute, January, 1939), by O. G. Patch, who was in charge of tests at Grand Coulee Dam. Mr. Patch gave 3750 and 6400 psi. as the minimum and maximum for the concrete control cylinders at Grand Coulee Dam for the month of September, 1937. This led the writer to state, with reference to the great refinement required in concrete mixing (*Journal*, American Concrete Institute, June, 1939, p. 180–187):

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[17] Consulting Engineer, New York, N. Y.

> "The concrete tests were made by a skilled crew which had been making tests daily on this job for two years. We find no comment on the 70 per cent spread in concrete strength, while at the same time the mixers were held to a spread of 10 per cent, which appears to have been due entirely to variations within the range of the test methods.
>
> "If concrete strengths spread 70 per cent in a month, in spite of unprecedented control, how important is it that each little grain of cement and sand be in exactly the right place?"

My analysis of the two tests of mixer efficiency then available showed a mean variation of about 1.0 per cent for three samples from a batch. Mr. Patch attributed the wide variation in strength to:

> ". . . cements from the different mills, and even from different runs of clinker from the same mill, differ 40 per cent or more in strength and because variations in aggregate grading are not entirely eliminated. . . ."

The same topics, tests of mixer efficiency and variation in concrete strength at Grand Coulee Dam, were further elaborated in separate papers presented at the Pittsburgh convention by writers of the U. S. Bureau of Reclamation. The first was covered by the paper on "Simplified Test for Evaluating the Effectiveness of Concrete Mixers," by Wing, Jones and Kennedy; the second was covered by the paper under discussion and revives the question that was answered by Mr. Patch: "What was the cause of the variations in concrete strength at Grand Coulee Dam?"

Instead of covering the first contract at Grand Coulee, the paper was based on two later contracts where cements of another type were used. The data are important for reasons entirely different from those stated by the authors. Few engineers are concerned with jobs that use a new type of cement in astronomical quantities that overstrain the resources of all the mills in a territory, but many will be interested to learn that cement and concrete tests do not follow the usual statistical relationships and that we can now begin to evaluate the errors of tests.

The authors state (p. 918):

> "Since most of the variations dealt with in the paper are large in comparison with experimental errors, the values given are without deductions for such errors unless otherwise specifically indicated."

My study of the paper shows:

A proper evaluation of test errors will make it necessary to discard most of the conclusions stated by the authors.

The wide variation in properties of cements resulted from a statistical study of about 4 per cent of the cements used on two large dams; the authors did not comment on the remarkable uniformity of about 7,000,000 bbl. of cement furnished by three mills.

Many of the special cements used were not made under normal operating conditions, hence probably none of the conclusions apply to standard cements.

The paper presents an erroneous picture of the cement for dam 2, due to an improper use of statistical methods.

The authors erroneously charged to the cements variations of mortar strengths resulting from outdoor temperature; atmospheric temperature, of course, is not a property of cement.

The paper presented contradictory data on concrete control tests made on Grand Coulee Dam for the month of July, 1937.

"Within-batch" variation is not a

measure of the errors of either laboratory or field tests of concrete; batch-to-batch variation must always be considered, and becomes of utmost importance in tests extending over many months.

The authors erroneously substituted the within-batch error as a measure of variation in all field tests of concrete, and entirely ignored batch-to-batch errors in computing variance in Table III.

Variation in concrete and cement tests increases as a logarithmic function of the number of tests; variation over a long period shows little more than the effect of number of tests.

The value v = 5.5 per cent was the within-batch error, and 7.3 per cent the all-tests error of carefully made laboratory tests of concrete.

The absurdly low coefficient of variation of field tests of concrete, 2.9 per cent, resulted from "twinning" of the duplicate tests at Grand Coulee Dam.

There was no proof that cement variation was responsible for a measurable portion of the over-all variation in field tests of concrete. If field tests had been made with the same accuracy as laboratory tests using a premixed cement, test errors would have obscured nearly all other causes of variation.

The variance due to field tests appears to be 6 to 15 times that claimed by the authors.

Owing to the large number of variables, strength tests of concrete are never in statistical control.

The two prediction equations for concrete strength given in the paper, have no rational basis and indicate absurd strengths for w/c above the narrow range covered by dams 1 and 2.

This discussion is devoted principally to a consideration of the experimental errors of the paper as compared with errors of other similar tests carried out by the U. S. Bureau of Reclamation.

VARIATION *Versus* GROWTH

The treatment of the paper is based on the usual meaning of variation, that is, "a measure of the dispersion of a group of values." It is unfortunate that *variation* gained a foothold in the nomenclature of statistics. In every-day English, *variation* is synonymous with *change*, but there are many types of *change* that are not *dispersions* and hence cannot be treated as *variations*. If *dispersion* had been adhered to, the absurdity of some erroneous applications of statistical methods would have been apparent. I use *growth* to mean a continuous change in the same direction—either positive or negative.

In applying statistical methods, it is necessary to keep in mind three factors:

1. The numerical data must be *homogeneous* with reference to the property under consideration.

2. The values must be capable of dispersion, and must actually show dispersion.

3. Differences due to well-defined causes must not be charged to *variation*; this term is properly applied only to the effect of a multiplicity of unknown causes.

Let us consider a few examples. If we make tension tests of 15 steel bars from the same lot or from the same type of steel from a mill, or on one bar each of the same type of steel from 15 different mills, we may compute the coefficient of variation. If we make tests on 15 bars in which the carbon content of the steel increased by uniform increments, it would be erroneous to compute a coefficient of variation. Instead of making 15 observations on the strength of the same steel, we are here making one observation each on 15 different steels of different compositions. We must be particularly cautious of computed variations where long periods of time are involved. Suppose we measured the height of a boy on each birthday and find that he grew 3 in. per year, from 30 in. at 4 yr. to 60 in. at 14. From these figures we can compute average height 45 in. and standard deviation 8.6 in. In some quarters this might be considered a legitimate problem in elementary statistical methods, but when applied to this boy's height it is nonsense, since there was no *dispersion*; his height was always normal and he was free to grow in one direction only.

There is another large group of cases in which a period of growth is followed (or preceded) by a more or less static condition to which statistical methods are sometimes mistakenly applied. Consider the case in the accompanying Fig. 1, where the vertical scale represents measured values at monthly intervals. The sloping portion defines a relationship that does not lend itself to statistical treatment; the horizontal portion defines another relationship entirely different from the first and shows that statistical treatment is unnecessary. If these were random observations we might treat them statistically, but there is only one chance in millions that a random selection of these fourteen values would fall in this order, and being a chronological sequence, *they cannot be random samples.* It would be erroneous to attempt to apply statistical methods to the data in Fig. 1.

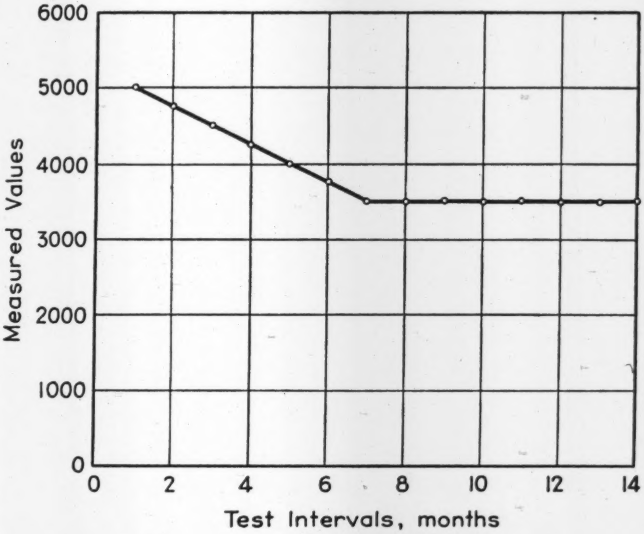

FIG. 1.—Statistical Methods Not Applicable to These Tests.

CEMENT FOR DAM 2

This cement is singled out for comment here for the reason that it illustrates a fundamental fault of the paper in drawing conclusions from statistical studies in cases where such methods are clearly not applicable.

The only information given on cement for dam 2 is contained in Table II and the curves at the right of Fig. 4. The following factors were omitted: within-bin variation of mortar strength, specific surface, C_3S, and heat of hydration. These values would have thrown much light on the variations of this cement as compared with the cements in dam 1.

The fineness of this cement steadily decreased from 2180 in September, 1940, to 1840 in August, 1941. This was not a variation, but a progressive "growth" in the properties of the cement. These changes in fineness were accurately reflected in the mortar strengths, but after August, 1941, the fineness was remarkably stable; this is reflected in both mortar and concrete tests made during this period.

It is natural for a new cement mill to begin by putting out the highest grade cement of which it is capable. It is also natural that the quality be gradually reduced to meet the acceptance standards of the purchaser. By lowered quality, I refer only to 28-day strengths; my own experimental studies showed that variations in concrete strength due to fineness of cement tend to disappear at later ages. At 5 yr. (the earliest age at which the most-stressed section of a large dam is likely to receive its full load), the difference due to fineness is one half that at 28 days; at later ages it becomes negligible

The high variations for concrete strength in dam 2 (and the unfavorable comparison with cements in dam 1) were largely due to:

Progressive lowering of fineness and progressive increase in water-cement ratio (and slump) during the first year of concreting,

Wide daily and monthly ranges of temperature.

None of these factors should be charged to *variation* in cement. If these causes were removed, the rating of this cement would probably have been much more favorable than the blended cements in dam 1.

A separate study should have been made of dam 2 for the period after August, 1941; this would give a true measure of variation of this cement as distinguished from the preceding months when it was undergoing a progressive reduction in fineness. I have made such a study using monthly averages scaled from the curves in Fig. 4. The specific surface can be taken from the diagram with considerable accuracy; for these 17 months, $v = 1.3$ per cent. If we double this value to take care of within-month variation, we have a 2.6 per cent which would be considered remarkably uniform.

Mortar strengths scaled from the curve are not so reliable, but my figures show for the same 17 months $v = 3.3$ per cent. The coefficients of variation of monthly means given in the paper and as computed by me are:

	Paper	Abrams
Specific surface..........	5.1	1.3
Cement strength.........	10.9	3.3

The values of the paper are three to four times as great as was actually shown by the cement for dam 2 *when we remove the element of progressive lowering of specific surface.* It seems likely that they were smaller than for any other cement.

Referring to Fig. 4 we find that the monthly average concrete strength for January, 1941 was 3800 psi. and declined rapidly until for July it was 2300, a loss of 1500 psi. The water-cement ratio increased during the same 6 months from 0.55 to 0.63, and specific surface dropped from 2100 to 1900. My figures show that the increase in water-cement ratio would account for about 800 psi. of the loss in concrete strength and the drop in fineness of cement would account for about 700 psi. The loss in strength of the concrete during these 6 months can be attributed wholly to changes in concrete proportions and fineness of cement. The effects of both of these factors on concrete strength have long been known. If the slump of concrete must be increased to meet the requirements of summer temperature, it should not be charged to the cement. These factors do not represent dispersions and should not be included in a study of cement variation.

The authors made the mistake of applying statistical methods to a cement that underwent a period of growth (lowered fineness) followed by a static period. This cement closely followed the pattern of the accompanying Fig. 1. It is erroneous to attribute to dispersion a progressive *change in the same direction* that extended over several months.

It should be noted that $v = 10$ per cent for mortar strength on dam 1 was only "estimated." My figures show that dam 2 cement from a single mill was more uniform than the blended cement for dam 1. This throws much doubt on the virtue of blending claimed by the authors.

v OF LABORATORY TESTS OF CONCRETE

In a paper on "Variations in Strength of Portland Cements . . ." the authors "begged the question" when they omitted a thorough consideration of test errors, and at the same time they ignored the most significant features of their data.

Concrete differs from most materials to which statistical methods are applied; we must not only make and record tests, but we must first manufacture the material to be tested. Concrete strength is affected by the quality and quantity of each of 4 to 8 different ingredients, and by a score of details of manipulation and environment. Variations in cement cover only a few of probably 25 variables that may affect the 28-day strength of concrete. The chance of getting the combinations that give both the highest and the lowest concrete strengths would be analagous to the chance of getting 25 heads *and* 25 tails in the first few throws of 25 coins; actually many million throws would probably be required before we get either 25 heads or 25 tails.

It can be accepted as axiomatic that it is impossible to secure a reliable measure of a variable that is smaller than the error of the method of measurement. One cannot weigh a dime on a truck scale. If 25 variables conspire to cause dispersion in concrete tests, it is obvious that a great many tests will be required before the limits of error can be established.

The evidence that I shall present to support my conclusion that the bulk of the errors that the authors attributed to cement and other causes should have been charged to tests, is found in various research reports of the U. S. Bureau of Reclamation, but my deductions are confirmed by many other investigations.

TABLE I.—VARIATION IN LABORATORY TESTS OF CONCRETE BY U. S. BUREAU OF RECLAMATION.

28-day compression tests of 6 by 12-in. cylinders.
Each group of tests was made on a single cement.
Sand and gravel used in all tests.
Wet-screened concrete.

Date	Source of Aggregate	Total		Approximate Strength, psi.	Coefficient of Variation, per cent			Authority[c]
		Batches	Tests		Two at a Time	Three at a Time	All Tests	
1932...	Boulder Dam	16	48	4000	2.8	2.7	8.0	371
1933...	Boulder Dam	9	27	3300	3.8[a]	4.7	6.9[b]	Manual
1935...	Parker Dam	4	12	4200	3.6	4.8	7.0[b]	509
1936...	Friant Dam	18	72	3300	2.7	3.0	...	...
1936...	Bartlett Dam	27	81	4200	3.1	4.4	...	557
1936...	Seminoe Dam	7	28	4300	2.7	3.1	...	576
Average				3900	3.1	3.8	7.3	
Corrected for number of tests					5.5	5.3	7.3	

[a] Average of using first 2 and last 2 tests of sets of 3. In other values in this column first 2 tests were used.
[b] Corrected for number of tests.
[c] Numbers are "*Technical Memoranda*" by U.S.B.R.

The accompanying Table I gives a résumé of six different groups of concrete tests carried out over a period of 5 yr. by the Denver Laboratory, in connection with investigations prior to the construction of large concrete dams. Each group of tests was made on a single cement; sand and gravel were used throughout; all concrete was wet-screened and tested at 28 days. These tests give a measure of the errors that are intrinsic to the most carefully controlled laboratory tests. We have computed the coefficients of variation for the specimens two or three at a time; in three instances we can compute "all tests" errors for batches made from the same materials, but at different times over periods of 4 to 8 days. At the bottom of the table the coefficients of variation are corrected for the number of tests.

The laboratory tests are quite conclusive in showing:

A remarkably close agreement among themselves.

A rapid rise in v as the number of tests increased.

Under the most careful control, using the same materials, repeat tests of concrete gave errors of:

2 cylinders from same batch..................	$v = 5.5$ per cent
All tests (12 to 48 tests)....	$v = 7.3$ per cent

The first group of tests in Table I was made in a study of laboratory control as a part of the concrete studies carried out prior to the construction of Boulder Dam (*Technical Memorandum 371*). The second group was used in illustrating strength variation of concrete in U.S.B.R. Concrete Manual, 1942.

The minimum test error of concretes made in the laboratory under the most careful control is much larger than that attributed by the authors to field tests. A further discussion of the significance of the number of laboratory tests will be presented after we have considered the variations of field tests.

v OF DUPLICATE TESTS OF CONCRETE FROM THE SAME BATCH AT GRAND COULEE DAM

Referring to Fig. 5 the paper states (p. 926):

> "The upper chart, showing within-batch control, was prepared by plotting for 34 batches of concrete sampled during a single month, the range in strength of two cylinders made from each batch. The mean range of 184 psi. corresponds to a coefficient of variation of 3.1 per cent. The fact that all plotted points came within the computed control limit and that the testing error was of the order of 3 to 4 per cent is evidence that mixing and testing procedures were well controlled."

It will be necessary to examine the above statement with utmost care, for several reasons:

This is the only statement in the paper of the actual basis on which errors of concrete tests were computed.

These tests assume unusual importance since the original data are available on which an independent computation of errors can be based—the only instance of its kind among thousands of tests referred to.

We are amazed that field tests gave much smaller errors than carefully controlled laboratory tests.

Table III shows that within-month errors were used in computing variance—here we have precise figures on what they were.

The tests in Fig. 5 were made at Grand Coulee Dam. This was the only large concrete dam under construction by the U. S. Bureau of Reclamation in July, 1937; besides the same tests were reported in detail by O. G. Patch in the paper mentioned above. In view of the fact that I had occasion to discuss these tests before, it may be well to give some of the background:

January, 1939, Mr. Patch gave a summary of concrete tests on Grand Coulee for September, 1937;

June, 1939, the writer commented on wide range of above tests;

September, 1939, Mr. Patch gave no further details of tests for September, 1937, but a complete schedule of tests for July, 1937;

September, 1939, the writer discussed tests for July, 1937 (*Journal*, American Concrete Institute).

Figure 5 of the present paper included selections from July, 1937, tests. This shows a striking partiality on the part of the U.S.B.R. writers for these tests, although similar tests were available for each of 45 months from December, 1935 to November, 1940.

Table II gives my analysis of the tests for July, 1937. Two cylinders were made from the same batch once during each of three daily shifts. I assumed that each set of three pairs represented one day's concreting. It is apparent that eight shifts were missed, or the tests for these shifts were omitted by Mr. Patch. However, my assumption does no violence to the data, since any random arrangement of the batches would give similar results. The table shows the difference and coefficient of variation of the two cylinders in a batch, daily averages and coefficients of variation, based on batch averages; weekly averages and coefficients of variation based on weekly averages; average and coefficients of variation for the month.

In the above quotation we have a definite estimate of the errors of test for one month as:

"mixing and testing procedure were well controlled,"
"coefficient of variation of 3.1 per cent," and
"the testing error was of the order of 3 to 4 per cent."

It will be seen from Table II and the following discussion that the actual tests for July, 1937, contradict nearly every feature of the statement quoted above. Mr. Patch gave 85 batches; we were not told the basis of the selection of the 34 batches used in Fig. 5.

Mixing.—My analysis in the *Journal* of the American Concrete Institute, 1939, showed that at Grand Coulee Dam mixing was held to an absurdly low mean variation of about 1.0 per cent, hence no question could possibly be raised with reference to the control of mixing procedure.

Two Reports on Same Tests.—The tests reported by Mr. Patch (Columns 2 and 3, Table II) differ in many respects from those plotted in Fig. 5:

	Patch	Fig. 5
Number of batches	85	34
Cylinder, max., psi	6650	...
Cylinder, min., psi	4440	...
Batch, max. (2 tests)	6542	6300
Batch, min. (2 tests)	4547	4550
Batch, avg. for month	5372	5300
Range, all cylinders, per cent	50	...
Range, all batches, per cent	44	39
Range, mean, psi	177	184
Batches with range		
300 psi. and over	14	5
400 psi. and over	6	1
500 psi. and over	5	1
100 psi. and under	30	7
v of batches (2 tests), per cent	2.9	3.1
v avg. daily (3 batches)	6.1	...
v avg. weekly (21 batches)	7.7	...
v avg. for month	7.2	7.2

It is impossible to reconcile these conflicting reports on the same group of tests. As treated by Mr. Patch the range from low to high cylinder was 50 per cent; the authors reduced this to 39 per cent by treating the average of 2 cylinders from a batch as a single test and by omitting the highest values. In spite of other wide discrepancies it is interesting to note that both give for the month $v = 7.2$ per cent. The authors obviously based some of their conclusions on the complete data as reported by Mr. Patch and others on the abbreviated report in Fig. 5.

The estimate of v of 3.1 per cent for concrete was based on the "within-batch" tests on two cylinders. (My

TABLE II.—VARIATION OF CONCRETE STRENGTH, GRAND COULEE DAM, JULY, 1937.

Tests by U. S. Bureau of Reclamation,
Columns 2 and 3, compressive strengths (psi.) of two 6 by 12-in. cylinders from the same batch, as reported by O. G. Patch, *Journal*, American Concrete Institute, June, 1939,
Columns 4 and 5, average and difference of the above,
Column 6, coefficient of variation (v) of two tests from same batch,
Columns 7 and 8, daily average strength and v based on three batch averages,
Columns 9 and 10, weekly average strength and v based on seven daily averages.

Day	For Batch					For Day		For Week	
	High	Low	Avg.	Diff.	v	Avg.	v	Avg.	v
1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*
1	5675	5535	5605	140	1.25				
	5830	5740	5785	90	0.78				
	5480	5400	5440	80	0.74	5610	2.6		
2	4785	4440	4612	345	3.74				
	5535	5305	5420	230	2.12				
	6050	5705	5877	345	2.94	5303	9.8		
3	5710	5630	5670	80	0.71				
	5975	5935	5955	40	0.34				
	5650	5055	5352	595	5.56	5659	4.3		
4	5905	5660	5782	245	2.12				
	5905	5730	5817	175	1.50				
	5250	5140	5195	110	1.06	5598	5.1		
5	5520	5130	5325	390	3.66				
	5625	5410	5517	215	1.95				
	5590	5500	5545	90	0.81	5462	1.8		
6	5560	5340	5450	220	2.02				
	5445	5340	5392	105	0.97				
	5860	5500	5680	360	3.17	5507	2.3		
7	5675	5675	5675	0	0				
	5280	5085	5182	195	1.88				
	5340	5225	5282	115	1.18	5379	4.0	5503	5.3
8	5750	5520	5635	230	2.05				
	5190	4985	5087	205	2.02				
	5475	5245	5360	230	2.15	5360	4.2		
9	4865	4775	4820	90	0.93				
	5500	5280	5390	220	2.04				
	5250	5215	5232	35	0.34	5147	4.7		
10	4640	4640	4640	0	0				
	5720	5340	5530	380	3.44				
	5295	5020	5157	275	2.67	5109	7.1		
11	5005	4680	4842	325	3.36				
	6160	5905	6032	255	2.11				
	5370	5260	5315	110	1.06	5396	9.1		
	5200	5145	5172	55	0.53				
	5305	5295	5300	10	0.09				
	5510	5320	5415	190	1.75	5295	1.9		
	5160	5055	5107	105	1.02				
	5110	4970	5040	140	1.39				
	4950	4720	4835	230	2.38	4994	2.3		
14	5395	5320	5357	75	0.70				
	5525	5200	5362	325	3.04				
	4720	4510	4615	210	2.28	5111	6.9	5202	6.3
15	5500	5480	5490	20	0.18				
	5305	4780	5042	525	5.31				
	5110	5090	5100	20	0.20	5210	3.8		
16	5260	5155	5207	105	1.01				
	4985	4820	4902	165	1.68				
	5520	5400	5460	120	1.10	5189	4.4		
17	5995	5695	5845	300	2.57				
	6235	5490	5862	745	6.35				
	5130	5040	5085	90	0.81	5597	6.5		
18	5800	5465	5632	335	2.96				
	5545	5445	5495	100	0.91				
	5740	5730	5735	10	0.09	5621	1.7		

TABLE II.—*Continued.*

Day	For Batch					For Day		For Week	
	High	Low	Avg.	Diff.	*v*	Avg.	*v*	Avg.	*v*
1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*
19	5170	5170	5170	0	0				
	4950	4790	4870	160	1.64				
	5820	5550	5685	270	2.38	5241	6.4		
20	5480	5340	5410	140	1.29				
	6650	6435	6532	215	1.65				
	5420	5320	5370	100	0.93	5774	9.4		
21	5020	5010	5015	10	0.10				
	5855	5855	5855	0	0				
	4605	4490	4547	115	1.27	5139	10.5	5396	8.1
22	5390	5370	5380	20	0.18				
	5835	5825	5830	10	0.08				
	4870	4680	4775	190	2.00	5328	8.1		
23	5710	5615	5662	95	0.84				
	6350	6240	6295	110	0.87				
	5320	5270	5295	50	0.47	5751	7.2		
24	5190	4915	5052	275	2.73				
	5685	5520	5602	165	1.47				
	5410	5305	5357	105	0.98	5337	4.2		
25	5305	4750	5027	555	5.50				
	5535	5340	5437	195	1.79				
	5530	5110	5320	420	3.95	5261	3.3		
26	5305	5005	5155	300	2.90				
	5625	5625	5625	0	0				
	5765	5625	5695	140	1.23	5491	4.4		
27	5305	5280	5292	25	0.24				
	5445	5385	5415	60	0.55				
	5130	4835	4982	295	2.96	5229	3.5		
28	5235	5145	5190	90	0.87				
	4765	4490	4627	275	2.98				
	6210	6155	6182	55	0.45	5333	12.0	5390	7.5
29	5720[a]	5640[a]	5680[a]	80[a]	0.70[a]				
Avg.	...	...	5372	177	1.66	5372	5.31	5372	6.8
v	7.3		7.2	...	2.9[b]	...	6.1[b]	...	7.7[b]

[a] Not used in averages or in computing *v*.
[b] Corrected for number of tests.

computations from the tests reported by Mr. Patch show this should be 2.9 per cent.) The questions then are:

Is the variation between two cylinders from the same batch a proper criterion of errors of concrete tests?

How do the errors of field tests at Grand Coulee Dam compare with carefully made laboratory tests by U.S.B.R. on concrete made from a premixed cement?

Is there a plausible explanation for $v = 2.9$ per cent?

Can we neglect batch-to-batch errors?

Are tests for a single month sufficient to establish variation?

When we pass from within-batch tests to the general problems of concrete tests we must include new causes of variation:

Different batches on same day,

Tests made on different days,

Tests by different men over long periods of time.

A mere catalog of the factors involved shows that the variation of duplicate tests from the same batch is not a proper measure of the error of concrete tests, whether made in the laboratory or in the field.

"Twinning" of Duplicate Tests at Grand Coulee Dam.—If duplicate tests made from the same batches under the

most careful laboratory control averaged v = 5.5 per cent, how did the authors arrive at v = 2.9 per cent for similar tests made at Grand Coulee Dam? An examination of the differences between duplicate tests from the same batch in the accompanying Table II shows unmistakable evidence of "twinning" of the strengths. There are many proofs of this "twinning":

1. The value v = 2.9 per cent is absurdly low when compared with 5.5 per cent for laboratory tests.

2. In the normal course there would be about 1 chance in 500 of finding a zero difference, but here we find five zero differences out of 85 batches. In 3 days (21st to 23rd) six out of nine differences were 50 psi. or less; there were 30 instances in which the range was 100 psi. or less.

3. The computed v for all cylinders was almost exactly the same (7.3 per cent) as for the batch averages (7.2 per cent). A normal distribution of errors would always give a considerably higher v for the individual values than after averaging the tests in groups of 2 or any other number.

4. There was pronounced "twinning" in the laboratory investigation in the Boulder Dam researches in the accompanying Table I. For the first two cylinders (in set of three) in 16 batches v = 5.0 per cent, for the last two cylinders, 3.4 per cent. The variance of the last two cylinders was less than half that of the first two.

The unmistakable "twinning" makes the tests at Grand Coulee Dam useless as a dependable basis for errors of field tests of concrete.

It is apparent that the within-batch errors were erroneously substituted for the within-month values throughout the paper as the sole measure of errors of field tests of concrete; this is shown by the "mixing and testing" variance of 20 in Table III (v = 4.5 per cent); the authors ignored entirely batch-to-batch errors and gave v = 0 for "month-to-month" variation, for both dams. We show below that the within-batch error is only a small part of the test errors that must be considered.

v of Concrete Tests Increases with Number of Tests

I discussed above the errors of laboratory tests and of duplicate tests made from the same batch at Grand Coulee Dam. I now present the more general case of errors of field tests. When we move from the laboratory to the field we encounter additional variables, some of which are:

Batching errors,
Size and grading of aggregates,
Variation in water-cement ratio,
Temperature of air and concrete,
Transporting and curing of specimens,
Tests by different men,
Night work,
Tests made over long periods of time, and
Variation in cement.

This catalog of additional variables leads us to expect much greater errors in field tests than under laboratory control where none of these variables enter.

The accompanying Table II gives the variation of field tests made at Grand Coulee Dam for July, 1937. The coefficients of variation given in the table were computed from batch averages (the method used in the paper), but I determined also the values using individual cylinders:

	Grand Coulee Dam		Denver Laboratory
	Batch	Cylinder	
Batch	2.9	2.9	5.5
Day	6.1	6.3	...
Week	7.7	7.2	...
All tests	7.2	7.3	7.3

Parallel values from the Denver laboratory tests from the accompanying

Table I are given for comparison. The Grand Coulee values were based on 168 tests, laboratory on 268 tests, so that the indications should be fairly reliable. A casual examination of the above table shows that a small number of tests does not represent the "universe" of concrete, but after having applied the usual correction factors the variation continues to increase as more tests are included.

These tests have been plotted by distinctive symbols in the accompanying Fig. 2. The four solid circles for the Denver Laboratory tests define a straight line to which we must give much weight when we consider that for 12, 27, and 48 tests the concrete was made at different times over a period of 4 yr. using different materials. A heavy line indicates the trend of the laboratory values; it can be represented by the expression:

$$v = 5.0 + 1.6 \log N$$

where N is the number of tests.

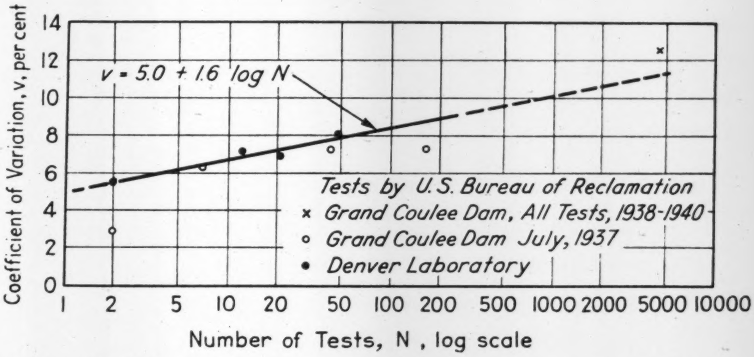

FIG. 2.—Variation of Concrete Strength Increased with Number of Tests.

An exponential relationship seems justified since previous experience has shown that a logarithmic function is the usual form for concrete properties, when the time element is involved. The number of tests is an indirect measure of time. The same principle will be seen in the log-time scales of Figs. 6, 7, and 8 of the paper.

No significance can be attached to within-batch errors unless it can be shown that they will not be affected by batch-to-batch errors. The accompanying Fig. 2 shows that even under the most careful laboratory control, 48 tests on the same concrete were probably insufficient to define errors of test.

In Fig. 2 we have also plotted (open circles) the values for v for 2, 6, 42, and 168 tests from Grand Coulee Dam, July, 1937, using the "all cylinder" variations. These values, in general, fall below the line for the laboratory tests. There seems to be two reasons: "twinning" of the duplicate tests, and 5400-psi. concrete as compared with 3900 in the laboratory tests. If we examine almost any series of tests in which concretes of different strengths were tested, we find that v is low for high-strength concrete and high for low-strength.

But the evidence is quite conclusive in showing that whatever the factors were that caused variation they were not very important when the errors of the field tests fall below carefully controlled laboratory tests. There were factors operating which would be expected to cause considerable variation. It will be recalled that Mr. Patch stated that "clinker from the same mill, differ 40 per cent or more in strength"; and it is known that the atmospheric temperature varied 50 F. or more during July, 1937. But these factors, separately or in combination with others, made no measurable impression on the variation of field tests of concrete, and in no case gave a higher v than was found in laboratory tests using a premixed cement and accurate control of water-cement ratio and temperature.

On dam 1 there were 4600 concrete tests made. If these tests were made with the same accuracy as laboratory tests, and the same logarithmic relation holds, we would expect "all tests," $v =$ 11.2 per cent. The accompanying Fig. 2 indicates that for 180 laboratory tests $v = 8.7$ per cent, hence we would expect month-to-month $v = (11.2^2 - 8.7^2)^{\frac{1}{2}} =$ 7.0 per cent; but this is the same as the value given by the authors (7.1 per cent in Table II). For "all tests" $v = 12.5$ per cent; the only portion of this that can be charged to causes other than tests is $(12.5^2 - 11.2^2)^{\frac{1}{2}} = 5.5$ per cent. This takes account of temperature, water-cement ratio, cement, and the probability that the test error was much higher than the value we assumed, based on laboratory tests. In view of the well-known effects of temperature and water-cement ratio, it seems doubtful whether any portion can be charged to cement.

If we attribute to dam 2 the same errors in field tests that were found in the laboratory, it appears that the high monthly mean, $v = 15.2$ per cent, can readily be accounted for by the progressive lowering of fineness of cement during the first year, violent changes in temperature (and the corresponding changes in water-cement ratio), without attributing any variation in concrete strength to the cement (other than progressive change in fineness).

v OF CEMENT TESTS

We have presented certain evidence on the rôle of experimental errors in laboratory and field tests of concrete. We shall now consider errors of cement tests.

Let us assume a bin of cement so thoroughly mixed that it is uniform in all parts with reference to both fineness and composition. If we make carefully controlled cement and concrete tests on random samples, we are justified in concluding that all variations are due to errors of testing and none to cement. It happens that cements B and C of the paper nearly conform to the assumed requirements. These cements are of special interest for many reasons:

They were quite similar, with nearly average strength, fineness, and C_3S,

Tests on duplicate samples from a bin were probably made on the same day,

The within-bin variations of specific surface and C_3S were negligible,

These cements were used throughout a period of 2 yr.,

They accounted for 65 per cent of the cements used in dam 1.

Compound Composition.—The average within-batch C_3S for cements B and C gave $v = 2.35$ per cent. Tests by U.S.B.R. on samples of a cement which had been "premixed at the plant" showed for both C_3S and C_2S, $v = 1.7$ per cent (*Technical Memorandum 371*, Boulder Dam Research). Cements B and C were sampled from the conveyor to storage, but the cement was turned over 4 times (or more) before it reached the blending stage; discharge to storage, loading to cars from storage, unloading from cars, and in discharging to blending conveyor; hence it is probable that when blended it was as uniform as the premixed cement. It is unfortunate that the authors did not investigate this factor by testing samples from the blending silos.

Specific Surface.—The average within-bin specific surface of cements B and C, $v = 2.9$ per cent, approaches the limit of error of the most careful repeat tests on the same cement. This belief is confirmed by repeat tests by U.S.B.R. (*Technical memorandum 371*) on ten lots of laboratory cements which gave an average minimum specific surface of 1780 and average maximum 1960, or a range of 7.4 per cent. If we take $v = \frac{1}{3}$ range, this gives $v = 2.5$ per cent, which is essentially the same as for cements B and C. We have no license to complain of variations that cannot be detected by the test methods.

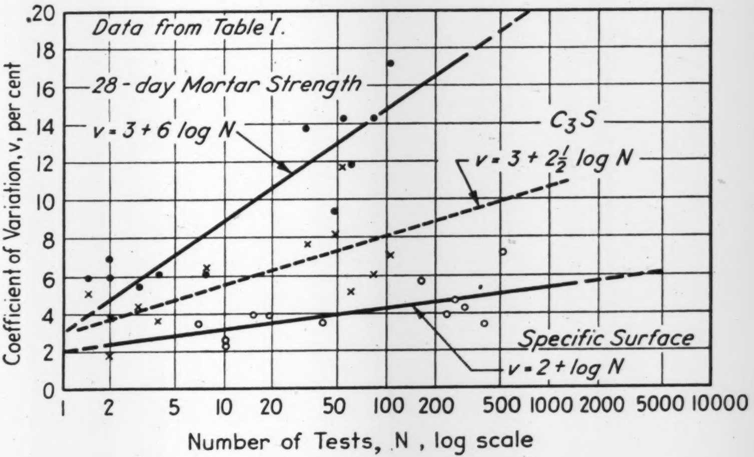

FIG. 3.—Variation of Cement Tests Increased with Number of Tests.

Following the pattern of the accompanying Fig. 2, we plotted in Fig. 3 the variations in specific surface against the number of tests, using both within-bin and all-tests values. The curve can be represented by the expression:

$$v = 2 + \log N.$$

For each cement there were five times as many tests for fineness as for C_3S or mortar strength. The diagram shows a relationship similar to that found for concrete tests; namely, variation increased as a logarithmic function of the number of tests.

C_3S.—Figure 3 shows the relationship between v and number of tests for C_3S; the curve may be represented by the expression:

$$v = 2 + 2\tfrac{1}{2} \log N.$$

Mortar Tests.—The authors admit (p. 924):

> ". . . the discovery that there was general correspondence between the cement strength-time curves and the outdoor temperature. . . ."

But outdoor temperature is not a property of cement; hence variation from this cause must be charged to testing.

The strength curves for cements B and C in Fig. 1 show what is practical in mill operation, and that the *actual variation* of these cements over a period of 19 months was extremely small. At first sight the curves seem to indicate that temperature had an important effect on strength, but we can eliminate some of this effect by making a study of the tests that fall in a narrow range of temperature. The writer made a study of the monthly means, scaled from the figure, that fall within the range 44 to 55 F. The coefficients of variation of 11 such strengths were 6.4 and 6.0, average 6.2 per cent. The eleven samples must cover the entire range of fineness and compound composition. If cements B and C had been blended in equal parts, the variation of monthly means would have been about $v = 4.4$ per cent. Table I gives for 28-day strength of cements B and C "all tests" $v = 13.0$ per cent; my analysis suggests that about 5 per cent was due to the temperature plus cements and 12 per cent to testing.

We saw above that the experimental error of laboratory tests of concrete increased with the number of tests. The accompanying Fig. 3 presents a study of 28-day mortar strength, using such data as can be gleaned from Table I of the paper. The general trend of the curve, based on both within-bin and all-tests variations, can be expressed by:

$$v = 3 + 6 \log N.$$

The constants in the expressions for cement variations are generally given in whole numbers, indicating that the formulas do not justify greater refinement. Values for one test in Figs. 2 and 3 have no significance; the curves were extended merely to show the origin of the first constants in the logarithmic expressions.

SUMMARY OF EFFECT OF NUMBER OF TESTS ON v

In every instance in which data are available v increased as a logarithmic function of the number of tests; since the number of tests was proportional to the amount of a cement used, there is a strong presumption that variation is not a characteristic of the cements, but rather of testing technique. This is especially true since concrete made of the same cement showed this relationship.

Following are the approximate variations of typical numbers of tests as read from the curves in Figs. 2 and 3; also certain values for field tests of concrete from dam 1:

Number of Tests	Specific Surface	C_3S	Mortar Strength	Concrete Strength	
				Laboratory	Field
2	2¼	3½	4½	5½	4½
3	2½	4	5½	6	...
10	3	5½	9	6½	...
30	3½	7	12	7½	...
100	4	8	15	8½	...
300	5	9½	18	9	...
1000	5½	10½	21	10	...
4600	6	12	25	11	12½

Probably the most striking feature of the above table is the small specific surface variation as compared with chemical analysis, mortar and concrete tests, where the human factor enters to a large degree. The human factor is largely absent in the turbidimeter test.

It will be noted also that the combined effects of specific surface and C_3S is almost exactly that of the variation in concrete strength. In the case of 100 tests (well within the experimental range) $(4^2 + 8^2)^{\frac{1}{2}} = 8.9$ per cent, would be the expected variation for concrete, if we assume that this variation was entirely due to these causes. The figure for concrete was 8½ per cent; however the laboratory tests of concrete on which it was based were made of premixed cements in which variations in specific surface and C_3S did not enter. This seems to be another proof that even the combined effects of specific surface and C_3S exert only a negligible influence as compared to that of tests.

The above table is striking in showing the extremely high variation of mortar tests that cannot be traced to either fineness of C_3S of cement, and are not reflected in the concrete; hence they are largely attributable to errors of tests. I think it is usual experience that mortar tests on a given cement are much more variable than laboratory tests of concrete. Apparently no tests whatever were made on these cements as they actually were used in the dams.

There is strong evidence that:

The usual correction factors for number of tests are too low when applied to cement and concrete.

Tests of cement and concrete requiring manual operations are never in statistical control.

The "all-tests" v of mortar or concrete have little significance except as a measure of errors of tests carried out over a long time.

VARIANCE OF FIELD TESTS OF CONCRETE

Throughout the paper variance due to field tests of concrete was based on a within-month value of v which appear to be in fact monthly averages of within-batch values. In the text it is stated that the within-month variation for a typical month (Grand Coulee Dam, July, 1937) was "of the order of 3 to 4 per cent," and gave 3.1 per cent as the exact value, (we saw above that this should have been 2.9 per cent). In Table III still another value, $v = 4.5$ per cent, was used for the within-month variation. The "within-bin" variations in the properties of cement were not reflected in the concrete on the dam.

Above we presented evidence that indicates that the all-tests variation for dam 1 was 11.2 per cent; this gave a variance of $11.2^2 = 125.4$, and quantitatively represents 80 per cent of the sum of all variances for dam 1. If we accept this value, the variance due to tests alone appears to be about six times that assigned to this cause. If we take the value $2.9^2 = 8.4$ as representative, the probable value was 15 times that assigned to this cause by the authors. It is apparent that the authors were in error in using the within-batch variation as a measure of variation of field tests and entirely ignoring batch-to-batch variation.

With the evidence before us, we are driven to the conclusion that there is no proof that cement variation was responsible for a measurable portion of the over-all variance of the strength of concrete cylinders on dams 1 and 2. If field tests had been made with the same accuracy as laboratory tests using a premixed cement, the test error would have obscured nearly all other causes of variation.

INFLUENCE OF COMPOUND COMPOSITION ON CEMENT STRENGTH

The paper states with reference to compound composition (p. 924):

> ". . . the reported differences in composition are decidedly inadequate as a means of explaining the large differences in mill strength levels."

In other words they now disown the principle that was developed in their Boulder Dam research. There are two explanations, either of which may account for the apparent failure of compound composition to reflect mortar strength. The composition of a blend cannot be computed by taking proportional parts of chemical analysis of the individual cements and computing a new composition. It seems likely that this error was made in the paper, since compound compositions were erroneously averaged in Tables I and IV. In *Technical Memorandum 371* we find that compound compositions of ten cements were averaged, although C_3S ranged from 12 to 54 per cent.

To illustrate my point let us take an extreme case, a blend of equal parts of a high-early-strength, $C_3S = 70.0$ per cent and a low-heat cement, $C_3S = 14.2$ per cent. The average is 42.1 per cent; but if we compute composition from the average chemical analysis

C_3S becomes 55.0 per cent, an increase of 30 per cent. In another instance of two low-heat cements the C_3S computed from the average chemical analysis was 7 per cent lower than the average of the cements. I have made no attempt to explore the extreme limits, but it is obvious that the compound composition computed from average chemical analysis is not the same as the average of the original compound compositions.

In the manufacture of low-heat cements many mills add iron ore to the raw mix. Should the manufacturer make a mistake and add the iron ore at the grinding mills instead of placing it in the raw mix for the kilns, the chemical analysis would show a low-heat cement, but the strength would be that of a modified cement.

CEMENT MILL CONTROL

A generation ago cement specifications generally contained a clause to the effect that the cement supplied must be manufactured in a mill that had been in successful operation for 5 yr., but since the bulk of the cements used in the United States during recent years was made in mills 25 to 40 yr. old, this clause disappeared from specifications.

It is unlikely that any of the mills supplying low-heat cement for dams 1 and 2 had ever made this type of cement before; it is apparent also that some of them did considerable experimenting, hence the cements were not made under normal operating conditions. The conclusions of the paper probably do not apply to standard cements made under normal conditions.

The gradual increase in strength of cement F from 4200 psi. in December, 1938, to 5800 in November, 1939, was a systematic "growth," the effect of which was reflected in the mortar tests; but the authors computed it as a variation.

Cement F was accepted and used on exactly the same basis as the other cements for dam 1 and was sold at the same price, although it had nearly twice the strength of some others. This shows a weakness in the method of specifying and using cement. Table I gives average 28-day mortar strength of cement F of 5000 psi.; Fig. 2 shows 4500 psi.

The authors seem to have been particularly unfortunate in their choice of cements for blending. The paper does a grave injustice to cements B and C by throwing them into a group of other cements that were not under control. We saw above that the cement for dam 2 was remarkably uniform after the fineness became stabilized. This makes it apparent that the problem of securing uniform cement from a single mill *can be solved*. These features were not discussed by the authors, although the instances cited covered about two thirds of the cements used in the two dams.

PREDICTION EQUATIONS FOR CONCRETE STRENGTH

Let us examine the equations at the bottom of Table II "concrete strength prediction equations" for dams 1 and 2:

$$\text{Concrete}_{28} = 10{,}790 - 14{,}600\ w/c + 0.38\ \text{Cement}_{28}.$$

$$\text{Concrete}_{28} = 7200 - 11{,}200\ w/c + 0.87\ \text{Cement}_{28}.$$

Taking the average cement strengths of 3250 and 2980 psi. we compute:

Water-Cement Ratio, by weight	28-day Concrete Strength, psi.		
	Dam 1	Dam 2	Abrams Dam 1
0	12 020	9790	14 000
0.3	7 650	6430	7 200
0.4	6 280	5310	5 700
0.5	4 720	4190	4 600
0.537	4 180	....	4 120
0.6	3 250	3070	3 700
0.7	1 820	2050	2 900
0.8	340	830	2 400
1.0	−2 580	−1410	1 500
1.2	−5 480	−3660	950

For narrow ranges of water-cement ratio used in dams 1 and 2 the formulas of the paper are not far off, but for w/c = 0.6 and over they give absurd values, as shown by a computed strength of minus 5480 and minus 3660 psi. for w/c = 1.2.

Twenty-five years ago the writer published an expression of the water-ratio strength law for concrete strength that can be used for prediction of concrete strengths where water-ratio varies. The general form was:

$$S = \frac{A}{B^{w/c}}$$

where:

S = 28-day concrete strength in pounds per square inch,

A and B = constants that depend on the strength level of the cement, and

w/c (an exponent) = water-cement ratio *by volume.*

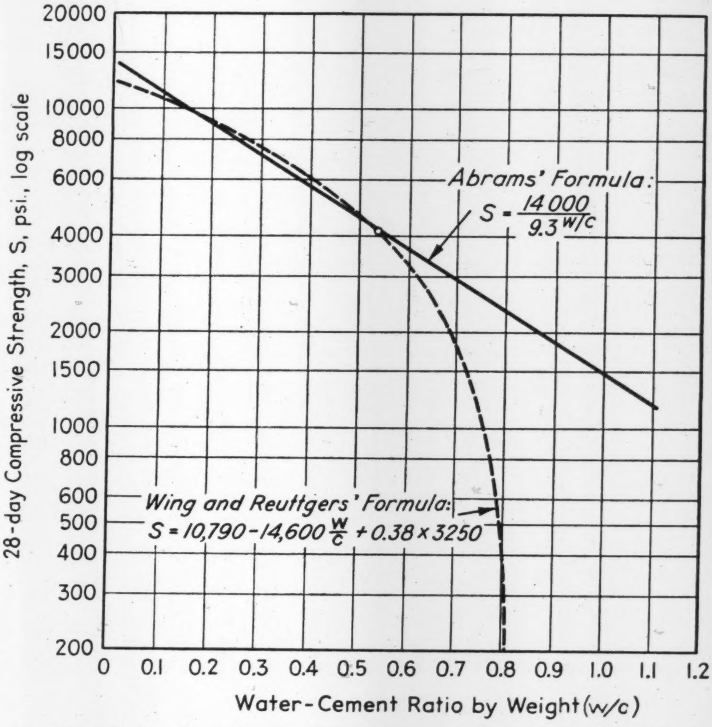

FIG. 4.—Prediction Formulas for 28-day Concrete Strength for Dam No. 1.

Studies made 1916 to 1918 showed A = 14,000 psi.; later studies indicated that 14,000 psi. was an invariant, a "ceiling" common to all portland cement concretes and represented the strength which all tend to approach, so long as they are cured under favorable conditions, but beyond which the strength cannot go when the specimens are molded under gravitational forces—that is, without consolidation by pressure.

B is a dimensionless constant that reflects the *quality* of the cement. The principal factors (aside from water-cement ratio) that affect the quality of cement (as measured by strength) are: composition, fineness, age, temperature, curing conditions. Many of these factors affect concrete strength in a way that one can be traded off against the other; for example, increased fineness can overcome lack of age. Age, temperature, and curing can be controlled during laboratory tests, so that the principle variables are fineness and composition of cement.

In 1916–1918 the cements used gave $B = 7$ (18.5 if w/c is by weight), however with increased fineness and improved compound composition B was gradually reduced until in 1942 (before the War Production Board ordered an inferior cement) the average standard portland cement (Type I) gave $B = 4$ (8 by weight). This means that for standard cements, concrete strength for a given water-cement ratio was nearly doubled in the 25 yr. between 1917 and 1942.

The above equation can be most easily visualized on a diagram, since it becomes a straight line if we plot water-cement ratio on direct scale and strengths to a log scale. Such a diagram is shown in the accompanying Fig. 4. In order to use the formula (or diagram) for predicting concrete strength all we need is one reliable value for the 28-day strength of plastic concrete made of the materials to be used and of a known water-cement ratio. In the table above I gave the predicted strengths for various water-cement ratios for dam 1. In order to arrive at these values, I used the first strength determined for this dam as scaled from Fig. 4, w/c = 0.54 (by weight) and strength 4200 psi. This gave a single point which is shown by an open circle in the accompanying Fig. 4. If we draw a straight line through this point and strength 14,000 psi., for w/c = 0, we have a plot of the equation:

$$S = \frac{14{,}000}{9.3^{w/c}}$$

This gives us a measure of the strengths to be expected from these materials with other water-cement ratios. The values can be read from the diagram. The starting point would usually be obtained from laboratory tests made prior to beginning the job, but I used the only value available to me.

On the accompanying Fig. 4 we show also the locus of the prediction equation of the paper for dam 1. It is, of course, impossible to show the negative values, since zero on this scale is at an infinite distance below the bottom of the figure.

The authors' prediction equations are meaningless for these reasons:

They cannot be used; such an equation can be derived only after the job is finished.

None of the constants used has a rational basis.

The formulas indicate that strength is a linear function of w/c, whereas it has been demonstrated by hundreds of series of tests that it is a curvilinear relation (on natural scale of strengths).

The narrow range of w/c makes it impossible to develop a workable equation.

The cement strength which enters into the formula is said to be a function of outdoor temperature at the point where tests were made.

It is unreasonable to expect the same type of cement to have 2.3 times as much effect on concrete strength on dam 2 as on dam 1.

Both formulas give absurd or impossible strengths for w/c which have been used in important structures.

No prediction formula can be expected to give precise results, since it is impossible to take account of such factors as grinding of cement in mixers and concrete temperature. That the formulas of the paper are not reliable is shown by the fact that Elephant Butte and Arrowrock Dams were built by the U.S.B.R. of concretes with w/c about 1.2, and the 28-day strengths were approximately that indicated by my formula (about

600 psi.); they did not have a strength of minus 5500 psi., as required by one of the formulas of the paper.

The principal conclusions were given at the beginning of this discussion.

MR. E. T. CARLSON[18] (*by letter*).—The National Bureau of Standards' Seattle laboratory which conducted the acceptance tests of the cement used in dam No. 1 has made a study of the test results, in an attempt to correlate the behavior of the various cements with their chemical composition and fineness. An account of this study may be of interest because of its relation to the paper under discussion. To facilitate comparison, the ten brands of cement used are designated, in the following presentation, by the same letters used by Wing and Ruettgers.

The study is confined to the variations in strengths of plastic mortar cubes at 7 and 28 days, together with the possible causes of these variations. Each set of specimens was made from a composite sample of cement representing, in general, 2000 bbl. The number of composite samples representing each brand varied from 1232 for brand C, down to 26 for brand H.

The first step in the analysis was to select the variables for consideration. The values obtained for mortar strength may be assumed to be functions of the following variables: (1) chemical composition; (2) fineness; (3) conditions of manufacture, such as fineness of the raw material, length and condition of clinker storage, and rate of cooling of clinker; and (4) variations in conditions or technique of testing. Under group (1) may be included the various analytical determinations, the four theoretical compounds calculated from the analysis, and any number of derived ratios and combinations, not all of which, of course, are independent of each other. Fineness, as represented by the Wagner turbidimeter test, is largely independent of composition, although this would not necessarily be true of particle size distribution. The factors included under group (3) cannot be evaluated mathematically and it is doubtful whether most of them remained constant for any given brand of cement. Under group (4) may be mentioned variations in technique of operators and in such conditions as temperature and humidity of laboratory and storage rooms. In addition to the systematic variations due to the causes listed, there is the normal statistical distribution to be expected of results of tests performed under controlled conditions. These non-assignable variations may be so large as to mask completely any effects due to composition and fineness but over a period of time it may be expected that they will tend to average out, permitting the systematic variations to be estimated.

According to currently accepted views, the early strength of portland cement is influenced by the tricalcium silicate (hereinafter abbreviated to C_3S). This constituent was therefore selected as one of the variables to be correlated with observed mortar strengths. Dicalcium silicate (C_2S) and C_3S are mutually complementary, their sum being relatively constant for a given brand, hence C_2S is not considered a separate variable. Tricalcium aluminate (C_3A) very probably would be found to affect the mortar strength, but due to the narrow range permitted by the specifications for this (theoretical) constituent, the percentage varied so little that it was considered advisable to neglect it. In most of the brands there was a fairly wide range in the calculated percentage of tetracalcium alumino-ferrite (C_4AF). Since this is directly proportional to the Fe_2O_3, the latter was selected as the variable representing the iron content, inasmuch as it involves no assumptions as to actual compounds present. The amount of added gypsum varied considerably, and the SO_3 content was taken as a third variable. This determination, of course, does not distinguish between the SO_3 derived from the gypsum and that present in the clinker. While it might be of interest to study the influence of free CaO upon strength, it was felt that the analytical determination lacked the requisite precision. Preliminary observations failed to disclose any marked relationship between MgO and strength, so this constituent was omitted from consideration, as was the ignition loss and the insoluble residue. The alkalies and other so-called minor constituents were not determined. Our chosen variables, then, were C_3S, Fe_2O_3, SO_3, and the specific surface.

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[18] Associate Chemist, Laboratory, National Bureau of Standards, Seattle, Wash.

A logical starting point in attacking the problem appeared to be the plotting of the 7-day and the 28-day strengths against the C_3S contents for all samples of a given brand. A typical plot of this kind is shown in the accompanying Fig. 5, for brand D. (C_3S was reported to the nearest per cent; some of the plotted points are displaced to right or left when there is no room on the line.) It is at once apparent that there is a definite trend toward higher strength with increasing C_3S. There is also a wide range in strengths for any given value of C_3S. One sample with $C_3S = 20$ has a higher 7-day strength than another with $C_3S = 31$. The relationship appears to be approximately linear over the limited composition range represented. To obtain the slope, the averaging was

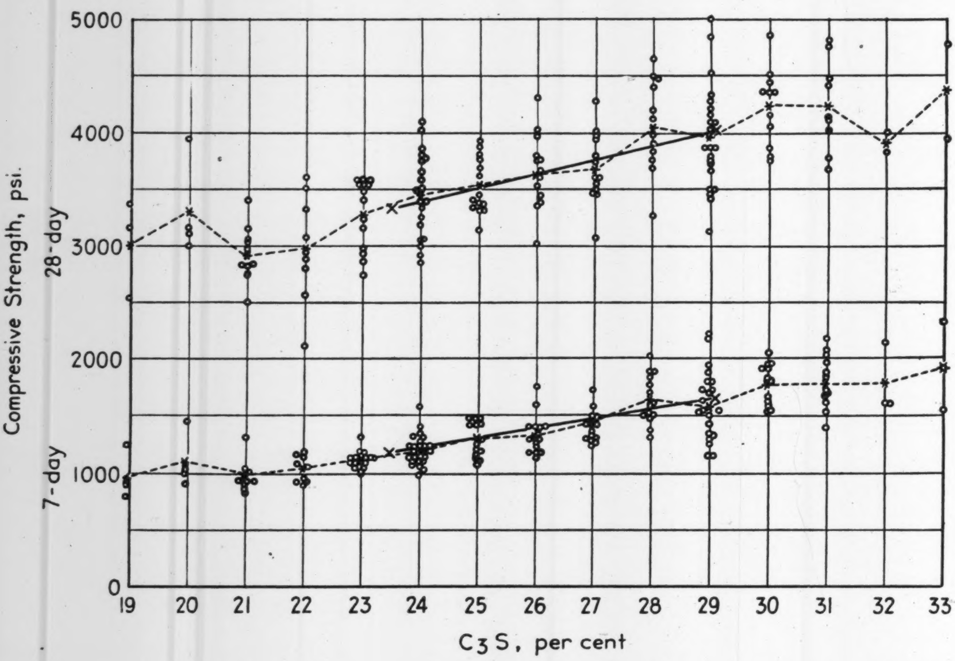

FIG. 5.—Compressive Strength *versus* C_3S, Brand D.

carried a step farther. The samples were separated into two groups, for convenience designated (a) and (b), the former including all samples with C_3S above the average for the brand, the latter all those with C_3S below average. The average of the 7-day strengths in each of the two groups was then computed, and likewise the 28-day strengths. The points thus determined were plotted in Fig. 5 and connected by solid lines, the slope of which represents the effect of C_3S on compressive strength for this brand.

So far we have assumed that groups (a) and (b) differed solely in C_3S content, but this is only approximately correct, as may be seen by reference to the accompanying Table III, showing the average specific surface, SO_3, and Fe_2O_3 for each of the groups in brand D. If it be assumed now that the spread in 7-day strength between groups (a) and (b) is due entirely to the combined effects of the spread in each of the four variables considered, factors may be assigned as follows: let t = the increase in 7-day strength due to an increase of 100 sq. cm. per g. in specific surface; c = the increase due to a rise of 1 per cent in C_3S; f and s, the increases due to 0.1 per cent Fe_2O_3 and SO_3, respectively, based on the total weight of the sample. An equation may now be set up, based on the figures in Table III:

$$-0.18t + 5.6c - 1.1f - 0.2s = 470 \text{ psi.}$$

TABLE III.—DIFFERENCE BETWEEN GROUPS (a) AND (b), BRAND D, IN COMPOSITION AND FINENESS.

	C_3S, per cent	SO_3, per cent	Fe_2O_3, per cent	Specific Surface	7-day Strength
Group (a).............	29.1	1.65	5.33	1946	1646
Group (b).............	23.5	1.67	5.44	1964	1176
Difference, (a) − (b)...	5.6	−0.02	−0.11	−18	470

Each of the other variables may now be taken in turn, the samples separated into similar (a) and (b) groups based on the particular variable under consideration, the averages for all four variables determined for each group, and equations set up, just as described above. Thus we eventually obtain four simultaneous equations containing the four variables, which may now be evaluated.

TABLE IV.—FACTORS AFFECTING MORTAR STRENGTHS (EXPRESSED IN POUNDS PER SQUARE INCH).

t = Increase in pounds per square inch due to increase of 100 sq. cm. per g. in specific surface.

c = Increase in pounds per square inch due to increase of 1 per cent C_3S.

f = Increase in pounds per square inch due to increase of 0.1 per cent F_2O_3.

s = Increase in pounds per square inch due to increase of 0.1 per cent SO_3.

Subscripts 7 and 28 refer to age at test.

Brand	Number of Samples	t_7	t_{28}	c_7	c_{28}	f_7	f_{28}	s_7	s_{28}
A.........	106	71	185	38	61	−40	−141	23	42
B.........	619	36	155	30	23	−21	−12	31	15
C.........	1232	34	123	34	38	−1	8	40	48
D.........	188	26	67	82	115	−20	−48	27	30
E.........	279	38	243	62	73	1	−23	26	33
F.........	168	156	460	38	86	8	13	34	100
G.........	122	15	215	49	58	−22	−43	15	93
H.........	26	5	102	62	72	−13	−27	−12	36
I.........	56	22	16	66	90	30	24	34	42
J.........	46	25	16	69	32	−6	25	73	97

TABLE V.—FACTORS AFFECTING MORTAR STRENGTHS (EXPRESSED AS PER CENT OF AVERAGE FOR BRAND).

(Symbols as in Table IV, but expressed in per cent rather than pounds per square inch.)

Brand	t_7	t_{28}	c_7	c_{28}	f_7	f_{28}	s_7	s_{28}
A..........	6.4	6.5	3.4	2.2	−3.6	−5.0	2.1	1.5
B..........	3.1	5.5	2.6	0.8	−1.8	−0.4	2.7	0.5
C..........	2.8	4.1	2.8	1.3	−0.1	0.3	3.3	1.6
D..........	1.9	1.8	5.9	3.2	−1.4	−1.3	1.9	0.8
E..........	3.2	6.1	5.2	1.8	0.1	−0.6	2.2	0.8
F..........	12.1	9.2	3.0	1.7	0.6	0.3	2.6	2.0
G..........	1.5	7.0	4.6	1.9	−2.1	−1.4	1.5	3.0
H..........	0.4	3.1	4.4	2.2	−0.9	−0.8	−0.9	1.1
I..........	1.7	0.4	5.1	2.5	2.3	0.7	2.6	1.1
J..........	2.0	0.5	5.6	0.7	−0.5	0.5	5.9	2.1

The factors obtained in this manner for the ten brands of cement are given in the accompanying Table IV, expressed in pounds per square inch. For purposes of comparison it is preferable to recalculate them on a percentage basis. Table V gives the same factors expressed as per cent of the average 7-day or 28-day strength of the indicated brand.

It should be emphasized that these factors have little quantitative significance. They may be considered rough approximations, decreasing in validity as the number of samples per brand decreases. With this in mind, the following facts may be noted.

The factors vary widely from brand to brand, probably, to some extent, reflecting differences in manufacturing procedure. The factors for C_3S for brands A and D, and for specific surface for brand A, may be compared with those obtained by Wing and Ruettgers by multiple correlation studies, the differences being no greater than would be expected, considering the difference in methods of calculation.

The values of t for all brands are positive, indicating that an increase in fineness is attended by an increase in compressive strength at both ages, within the range represented. (The average specific surface for the several brands ranged from 1888 to 2016 sq. cm. per g.) The wide variance in this factor between brands may be due in part to the inadequacy of the Wagner turbidimeter to measure the surface of the finer particles, mentioned by Mr. Tucker in his discussion.

All values for c are also positive, and the factor for the 28-day strength is in all cases lower than the corresponding one for the 7-day, on a percentage basis. This is to be expected, since the C_2S probably also contributes to the 28-day strength, and this constituent normally increases as the C_3S is lowered. The spread in the values for this factor is no doubt due in part to the fact that the brands vary in glass content, causing a variable error in the compound calculation.

With the exception of brand H, represented by only 26 test samples, all the values for s are positive. An increase of 0.1 per cent in SO_3 apparently results in an increase in strength, especially at 7 days, in some cases comparable with the increase due to 1 per cent additional C_3S.

No such uniformity is observed in the values of f, which are sometimes positive, more often negative. These figures are in general considered less significant than those for t, c, and s, since in several cases they are based on a relatively small spread in Fe_2O_3, increasing the relative weight of accidental variations.

The factors given in the accompanying Tables IV and V cannot be expected to be valid over a range of composition and fineness greater than that represented by the samples under consideration. Within this range, however, and to the extent that our assumptions are correct, they may be used to calculate the expected deviation of the strength of any given sample from the average for that brand. Any difference between the strength thus calculated and that actually observed for the same sample may then be attributed either to changes in manufacturing procedure, or to changes in the testing conditions or technique, or to nonassignable variations.

We are now in a position to determine whether these differences between calculated and observed strengths show any systematic trend from month to month. For this purpose, instead of computing the deviations for each sample tested, it is far easier and equally instructive to work on the basis of monthly averages. For each brand, the samples taken during a given month were grouped, and the average specific surface, C_3S, Fe_2O_3, and SO_3 determined. With the aid of the factors listed above and the average 7-day strength for the brand as a whole, the theoretical average strength for the month was calculated, also the difference between this calculated value and the actual average strength for the month. These deviations are plotted, month by month, in the accompanying Fig. 6. Only four brands are represented, as the others were used intermittently or for a brief period only.

As would be expected, the deviations show marked irregularities, especially during periods when few samples were being tested. However, all four curves show a drop from March to April, 1939, followed by a peak in June or July, a decline to October or November, and a subsequent rise. It is believed that this parallel behavior is rather too much to attribute to chance, but it would be extremely difficult to assign a reason for the trends. Laboratory conditions were well controlled, as has been mentioned by Wing and Ruettgers, but there may have been slight, unnoticed changes in operational technique. On the other hand, it is known that at least one manufacturing procedure changed somewhat during this period, as the mills had accumulated large clinker piles, more or less exposed to the weather, and these were gradually exhausted during the summer and autumn. It is likely that other manufacturing procedures may also have changed.

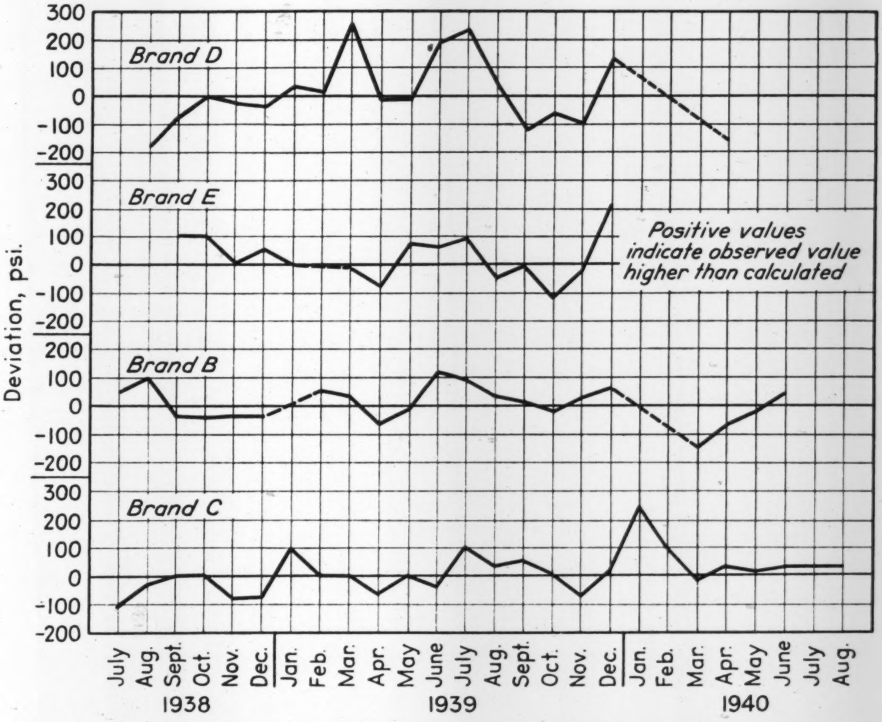

FIG. 6.—Deviation of Observed 7-day Strength from Calculated (Monthly Averages).

MESSRS. S. P. WING [19] AND ARTHUR RUETTGERS[19] (*authors' closure*).—The paper has evoked favorable and unfavorable comments from various quarters. The authors are appreciative of both, realizing that in their initial attempt to apply the statistical methods outlined in the A.S.T.M. Manual[20] and in a few excellent but rigorous papers in the *Proceedings* of the Society[21] they probably failed to strike the best balance between the exactness desired by the skilled statistician and the clarity expected by the readers of cement and concrete literature to many of whom statistical concepts and terminology are still a mystery.

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[19] Engineer and Senior Engineer, respectively, U. S. Bureau of Reclamation, Denver, Colo.

[20] A.S.T.M. Manual on Presentation of Data (April, 1943). (Issued as a separate publication.)

Mr. Kinney raised a pertinent question in inquiring (1) whether the reported variations in the low-heat cements purchased for dams Nos. 1 and 2 might not be greater than normal, due to previous inexperience of the mills in the manufacture of low-heat cement, and (2) whether the tests on early samples were found to show greater variations than later ones. The discussions of Messrs. Bates and Abrams bear on the first part of Mr. Kinney's question—Mr. Bates taking the position that the variations were not abnormal and might even be less than for standard cements, and Mr. Abrams making the sweeping assertion that "many of the special cements used were not made under normal operating conditions, hence probably none of the conclusions apply to standard cements." The authors, in the light of the discussions presented and of other studies made, are willing to venture a step further than they did in the paper, by stating that for single mills the variations reported are reasonably representative for any type of portland cement produced over a period of a year or more under similar specification requirements, but that between mills the variations are probably greater for low-heat cements than for standard or modified cements. In this connection it is of interest that "within bin" and "all tests" coefficients of variation for modified cements supplied by mills A to F inclusive for dam No. 1 were found to be substantially the same as those reported for low-heat cements in Table I. The variation between mills, however, was less for the modified cements.

The authors are unable to supply a positive answer to the second part of Mr. Kinney's question, as considerable work would have been required to obtain such an answer. However, a cursory examination of the acceptance test records failed to disclose any marked difference between the variations applying to the initial and subsequent periods of low-heat cement production.

Messrs. Hanna, Tucker, and Carlson touch on the possible inadequacy of the Wagner turbidimeter for measuring cement fineness and cite this possibility as a factor contributing to the reported variations or entering into their reconciliation. The Blaine or Lea-Nurse air-permeability form of apparatus is suggested as a promising substitute.

From tests made in the Denver laboratory of the Bureau of Reclamation, in which the Wagner and Lea-Nurse instruments were directly compared, it would appear (see accompanying Fig. 7 (*a*)) that for a given brand and type of cement ground to different finenesses in the same way, the Lea-Nurse (and probably the Blaine) instrument would provide no better relative index of variations in specific surface than the Wagner. In other words, had the acceptance tests been based on the air-permeability method, the within-mill picture of variations in cement fineness probably would not have been altered appreciably unless differences in grinding procedure were involved. One especially interesting feature of the tests is that companion samples of cement, treated by stirring in a mortar with a rubber pestle to break up flakes without imparting a grinding action, showed substantially higher test values but the same straight-line relation between the two instruments as the untreated samples. On the other hand, tests made on 17 different cements (see Fig. 7 (*b*)) showed a significant lack of concordance between the two methods of measurement. Hence, while the Denver data do not indicate which instrument gives a more nearly true measure of specific surface, they do not deny the possibility that the air-permeability method is superior and that its use in the acceptance tests for dam No. 1 cement might have helped materially to explain the large differences in mill strength levels. Further comparative tests with the two instruments may also help to explain why the Wagner fineness determinations are not more closely associated with water requirements and cement strengths for different grinding procedures, as commented on by Messrs. Tucker and Stadtfeld.

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[21] H. W. Leavitt and H. A. Pratt, "A Statistical Analysis of Compression Tests on Mortar Cylinders, Cubes and Prisms," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 851 (1939).
H. W. Leavitt, "A Study of Plastic Mortar Cubes," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part I, p. 321 (1937).

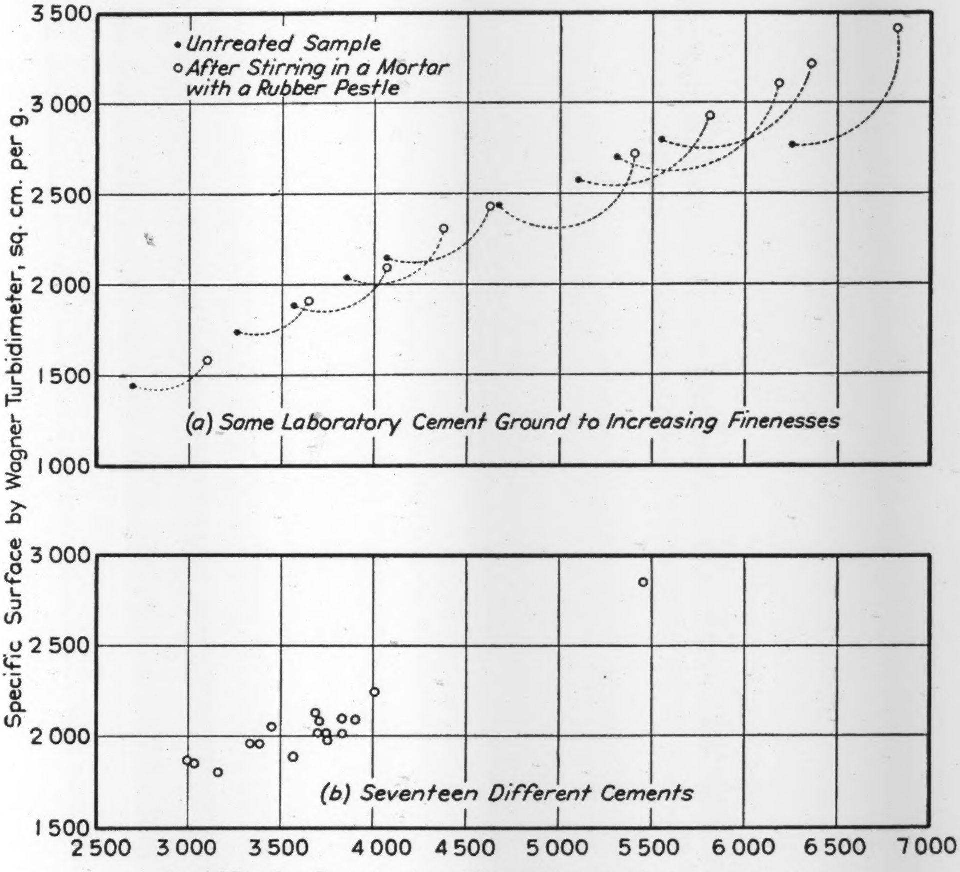

FIG. 7.—Comparison of Specific Surface Determinations by Wagner Turbidimeter and Lea-Nurse Air-Permeability Apparatus.

Cement composition, in addition to

specific surface, was accorded an active place in the discussions. The authors are particularly grateful to Mr. Carlson for his clear-cut analysis of unit contributions to cement strength for the ten cements furnished for dam No. 1. Mr. Carlson's findings and his constructive comments should convince everyone of the futility of attempting to charge the cement variations reported in the paper to cumulative testing errors and in this sense is a reply to Mr. Abrams' comments. The accompanying Fig. 8 was prepared by the authors to show graphically the nature

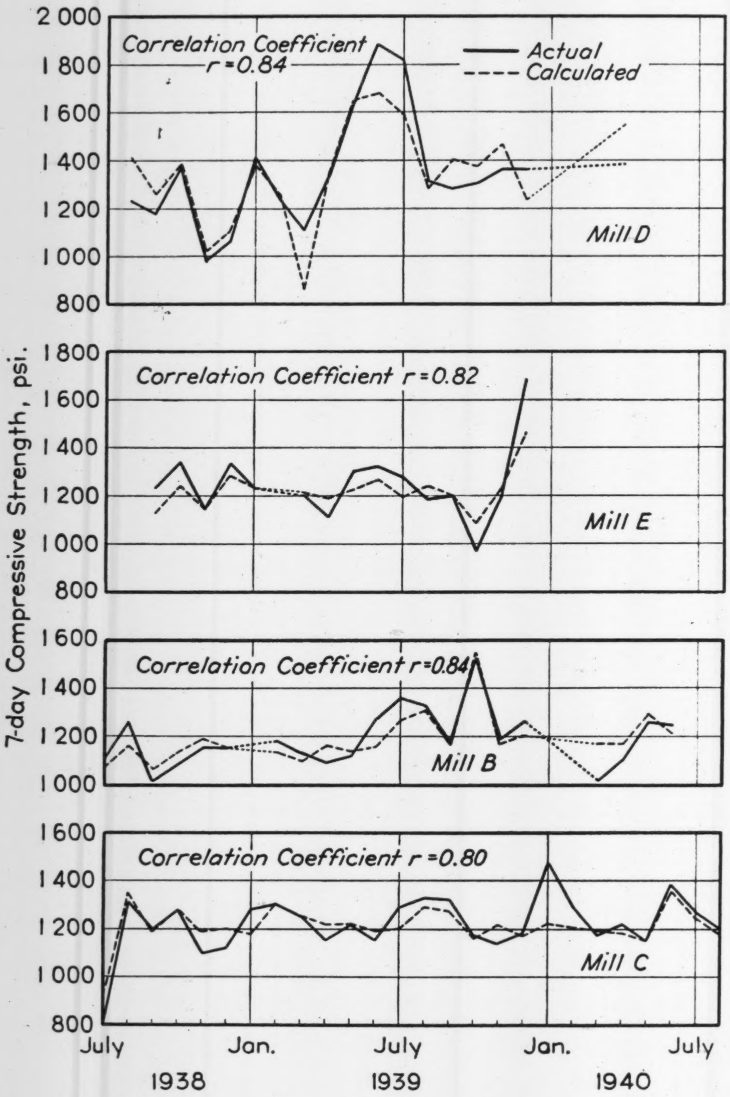

FIG. 8.—Comparison of Actual and Carlson's Calculated Cement Strengths for Dam No. 1.

of the agreement between the observed mean monthly mortar strengths at 7 days for cements B, C, D, and E and Mr. Carlson's calculated strengths. The authors also derived the corresponding correlation coefficients and found them to be 84, 80, 84, and 82 per cent, respectively, with an average of 82.5 per cent, demonstrating that the variations in the four selected factors, namely, specific surface, C_3S, Fe_2O_3, and SO_3 accounted for about two thirds of the total strength variance. It is gratifying that Mr. Carlson, in his own unique way, confirmed the authors' suspicion that some systematic influence was operating, particularly during 1939, to cause the various cements to show similar time-strength trends. This confirmation is the more significant because Mr. Carlson's analysis pertained to 7-day strength and that of the authors to 28-day strength. Now that the presence of the phenomenon has been reasonably established but the underlying cause has not, the matter might well merit the future attention of testing and research agencies such as the National Bureau of Standards.

Mr. Tucker points out, appropriately so, that correction of cement composition for glass content probably would have resulted in closer correlation with strength. Unfortunately, data needed to make the correction were not available.

Mr. Abrams criticizes the method used by the authors for obtaining average compound compositions and gives an example purporting to show that the average compound composition differs, depending on whether it is obtained by averaging the computed compounds of the individual cements or by calculating a compound composition from the average oxide analysis. The authors take issue with Mr. Abrams on this important point by stating that as long as the same compounds are represented in all cements averaged, the same result will be obtained by either method of calculation if properly executed. Had Mr. Abrams supplied sufficient detailed information, the error of his procedure or calculations might have been pointed out.

Mr. Stadtfeld makes some interesting comments with respect to the recent cement specifications of the Board of Water Supply of the City of New York and the experiences therewith. If the information on cement manufacturing procedure obtainable under such specifications had been available to the authors for the cements reported in the paper, undoubtedly there would have been less need for speculation as to the causes of some of the variations. However, the authors do not share the apparent view of Mr. Stadtfeld that the use of similar specifications would have eliminated the need for or impaired the utility of an analysis of cement variations and their significance. They regret that Mr. Stadtfeld did not supply, and they were unable to find in the literature, any quantitative data from which the claimed superiority in uniformity and general quality of the Board of Water Supply Cements might be judged. A search of the literature did disclose some surprising data[22] on the variations in 28-day compressive strength of 6 by 12-in. concrete control cylinders applying to six tunnel-lining contracts for the Delaware Aqueduct, the concreting period in each case being about one year. From one to three brands of cement were used on a single contract, and the number of test cylinders ranged from 51 to 138 per contract, with an average of 83. The surprising feature is that whereas the average strengths for the six contracts are very uniform (4876 min. to 5260 psi. max.) but quite low (for 1½-in. max. size aggregate and a cement content of 2 bbl. per cu. yd. of concrete), the coefficients of variation in cylinder strength as derived by the authors range from 13 per cent to 26 per cent, and average 19 per cent. Moreover, the highest coefficient of variation (26 per cent) applied to the contract on which but one brand of cement and only natural aggregates were used. With such relatively high variations in control cylinder strengths, it appears either that cement strength variation was a prominent factor or that the high quality cement may have been jeopardized in the final concrete product by permitting wide fluctuations in water-cement ratio, assuming of course that reasonable care was exercised in the fabrication, storage, and testing of the cylinders.

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[22] "Concrete Lining of Tunnel in the Eastern Department," *The Delaware Water Supply News No. 105*, December 1, 1942.

With regard to cement blending, Mr. Stadtfeld seems to attach greater importance to "tracing back failures in a structure," by maintaining the identity of each cement in the concrete, than to possibly minimizing the occurrence of failures through cement blending and the greater control of concreting operations associated therewith.

The discussion by Mr. Abrams contains so many contradictory, irrelevant, and inaccurate or unreasonable statements that any point by point rebuttal would, in the opinion of the authors, be inappropriate as well as inconsiderate of the readers. The authors are therefore confining their further comments to a few of the major questions, and particularly those of general interest. One of these is concerned with that elusive term "experimental error."

Mr. Abrams' principal argument, in his criticism of the authors' analyses of cement variations, is based on plotting "number of tests" (on a log scale) against a heterogeneous assembly of "coefficients of variation" (on a linear scale) which he ascribes to errors of laboratory and field tests, and then making a wide extrapolation of the data to arrive at an estimate of the over-all experimental error for a given total number of tests. A comparison of the estimated and reported values is then used to arrive at an estimate of the variations chargeable to cement, water-cement ratio, etc. The principal defect of Mr. Abrams' analysis, aside from the use of data of large inherent error (and sometimes the misuse of data, as subsequently illustrated), lies in the fact that the increasing coefficients of variation plotted against increasing numbers of tests (actually increasing periods of time) include to a greater and greater degree the effects of the specific variables, such as cement and water-cement ratio, which the analysis purports to exclude and finally to evaluate.

The method of analysis used in the paper to apportion the variations in concrete strength chargeable to cement and other factors is far more discriminating than Mr. Abrams' method, and the direct opposite in approach. Various correlation studies were first made of the cement acceptance test data, the field concrete control data, and the tie-in of the two. The results of these studies then made it possible, in preparing the table of variances in field cylinder strengths (Table III), to derive and deduct from the computed total "within-month" and "month-to-month" variances the values directly chargeable to cement, water-cement ratio, and contrete temperature. Prior studies also permitted making a reasonable deduction for "within-month" experimental error for mixing and testing, including within-batch and batch-to-batch errors. Thus the variance values remaining—listed as "unaccounted for"—were available to take care of non-assignable causes of variation, including "month-to-month" experimental error on which the authors had too little information to base a reliable estimate. The totals of the variances listed as "experimental error" and "unaccounted for," namely, 60 for dam No. 1 and 73 for dam No. 2, correspond to coefficients of variation of 7.7 and 8.5 per cent, respectively. With the above supplemental comments the authors trust it is clear that their analysis of variance is fundamentally sound and that Mr. Abrams' reiterated charges that batch-to-batch errors were ignored are unjustified. The authors do acknowledge and accept the responsibility for their loose use of the term "experimental error," criticized by Mr. Tucker, and for not making it clear in the paper that the omission of an allowance for "month-to-month" experimental error in Table III was intentional, for the reason stated.

An example of the misuse of data by Mr. Abrams will serve to demonstrate further the unreliability of some of his deductions. In his endeavor to show that the reported "within-bin" coefficients of variation in specific surface for cements from mills B and C (see Table I) are comparable with the error of repeat tests on companion samples of cements, he cites data taken from Bureau of Reclamation *Technical Memorandum No. 371.* Investigation discloses that the difference between the quoted specific surfaces of 1960 and 1780 sq. cm. per g. is not the average error of repeat tests by the Bureau of Reclamation on ten lots of laboratory cements, as alleged. Instead it is the average within-bin range in specific surface from regular acceptance tests made by the Bureau of Standards on 9 bins of cement averaging 33 test

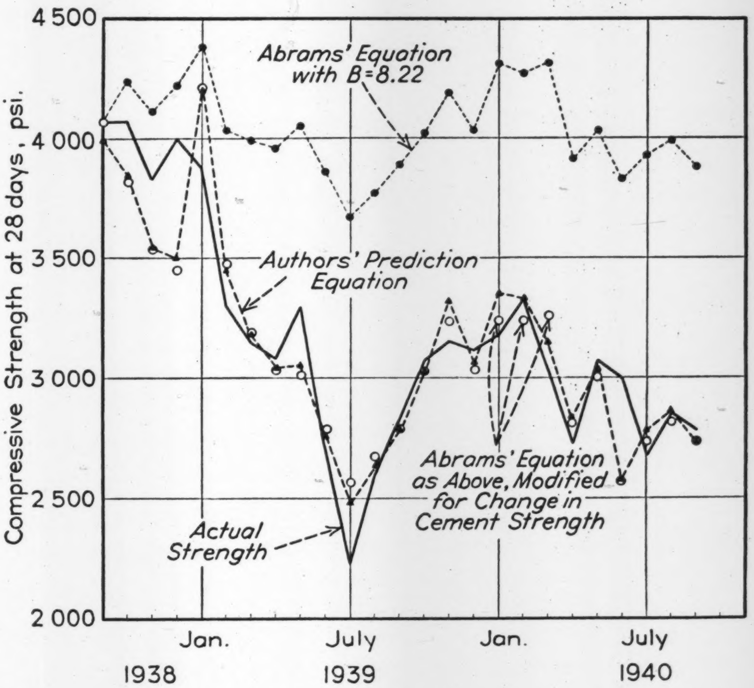

FIG. 9.—Comparison of Actual and Predicted Strengths of Concrete Control Cylinders for Dam No. 2.

samples per bin. If this range of 180 sq. cm. per g., or 9.6 per cent (incorrectly reported as 7.4 per cent by Mr. Abrams) were reduced to a within-bin coefficient of variation, the value would compare favorably with others reported by the authors in Table I.

Mr. Abrams' criticism of the use of statistical methods in analyzing the variations in cement and concrete for dam No. 2, on the ground that the test data are not homogeneous, is entirely out of line with the authors' interpretation of the A.S.T.M. Manual,[20] their principal guide. It is significant that of the nine commentators, including such authorities on statistics as Mr. Leavitt, only Mr. Abrams criticized the paper in this particular.

One other point of interest raised by Mr. Abrams has to do with the concrete strength prediction equations presented by the authors (see Table II) primarily as a means of conveying to the average reader, in readily understandable form, the regression coefficients resulting from their multiple correlations of concrete strength as a function of water-cement ratio and cement strength for dams Nos. 1 and 2. It seems hardly necessary to add that the equations were not expected to replace the Abrams water-cement ratio law equation. On the other hand, any attempt to predict concrete strengths solely on the basis of variations in water-cement ratio is bound to give unsatisfactory results when the cement strength is also varying appreciably. This is shown in the accompanying Fig. 9 by the large disparity between the reported control cylinder strengths for dam No. 2 and the prediction values from the Abrams equation, as compared with the prediction values from the authors' equation. The disparity, however, is virtually removed, as shown in the figure, by adjusting the Abrams prediction values by the gain or loss in the cement acceptance test strengths with respect to the strength applying to the first month. This finding leads the authors to suggest that the utility of the Abrams formula might be extended, for large jobs having access to mortar-cube cement-strength test results, by expanding the equation to the form: $S = \frac{14{,}000}{B^{w/c}}$ plus gain (or minus loss) in cement acceptance cube strength, the value of B to be fixed as soon as the concrete cylinder strengths and corresponding water-cement ratios have been reasonably established. An equation of this form could apparently be applied to strength tests at any age, so long as the test ages of the concrete and cement specimens conformed.

Mr. Tucker cites a second probable reason for the large disparity in reported variations in strength of the 22-in. and 6-in. diameter drill cores taken from dam No. 1. The authors recognize the logic of the theory that the coefficient of variation in strength of a given concrete is a function of the cross-sectional area of the test specimen, although they have found no experimental data of their own, other than the core data in the paper, to support the position that the effect is as large as represented by the inverse ratio of cylinder diameters.

With regard to Mr. Freeman's discussion, the authors appreciate fully the difficulty in finding means or incentive to analyze rapidly accumulating test data during periods of heavy construction activity, particularly when the tests are primarily for routine control and their immediate purpose has been served. Nevertheless, it is the growing conviction of the authors that if testing organizations in general would allot a larger share of their time and resources to analyzing their data more thoroughly, with an eye not only to discovering their true meaning but to effecting improvements in test planning and procedure, the over-all costs would be less and the benefits greater.

The authors are grateful for and encouraged by Mr. Leavitt's favorable reactions. His comparison of coefficients of variation in strength of cement and concrete from data in reports of the Maine Technology Experiment Station with those in the paper prompted the compilation of the comparative data in the accompanying Table VI. The table gives the coefficients of variation in 28-day strength of concrete control cylinders for a number of construction jobs of four months' minimum duration, including those referred to in the paper and discussions. Items Nos. 1 (Denver Laboratory) and 20 (Joint Committee Specifications, 1940) were incorporated in the table to aid the reader in judging, qualitatively and quantitatively, the coefficients of variation for the 25 construction jobs.

The Denver Laboratory value of 5.8 per cent (corrected for slight variations in water-cement ratio) applies to a long series of control tests in which it was aimed to produce concrete of constant slump with the same cement and aggregates in fixed proportions, the cement being obtained from periodic laboratory grinds of equal proportions of three stocked clinkers of normal composition. Inasmuch as there were three test cylinders per batch of concrete and the tests extended over a period of about 20 months, the coefficient of variation in strength includes not only the effects of within-batch and batch-to-batch variations but also of within-month and month-to-month variations. In other words, the coefficient represents an upper limit of long-time experimental error for well-controlled concrete strength tests.

The Joint Committee Specifications[23] indirectly provide a monetary incentive for the production of uniform concrete by defining failure to meet specification requirements as that which obtains when the average strength for 1 day (3 cylinders), 2 days (6 cylinders), 3 days (9 cylinders), and 5 or more days (15 cylinders) consecutive placing of concrete is less than 85, 90, 95, and 100 per cent, respectively, of the specified minimum allowable strength assumed for design. These specifications can be met, under controlled conditions, with a probability of avoiding failure on any count 43 times out of 44 by producing concrete with an average strength about 10 per cent in excess of the specified strength, if the coefficient of variation in cylinder strengths is kept within 20 per cent—the value listed as item 20 in Table VI. As seen from the table, about a fourth of the jobs did not control their concrete to this degree of uniformity. In such cases the alternative to securing better control and still have the same security against specification infringement is to increase the margin between average strength and specified strength. For a design strength of say 2000 psi. and a coefficient of variation of 38 per cent (the highest in Table VI), this would require a strength margin of about 50 per cent or an average strength of 3000 psi., instead of 2000 plus 10 per cent or 2200 psi.

An assembly of field test data similar to that in the accompanying Table VI but based on mean variation instead of coefficient of variation was included in a paper by Roderick B. Young[24] nearly 20 yr. ago. In commenting on what the variation should be for a well-conducted job, Mr. Young stated:

"The writer believes that on work

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[23] Report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete Submitting "Recommended Practice and Standard Specifications for Concrete and Reinforced Concrete," Am. Soc. Testing Mats., June, 1940. (Issued as a separate publication.) Data are computed from par. 327-SA, p. 113.

[24] Roderick B. Young, "Seven Years of Experience with Job Control of the Quality of Concrete," *Proceedings*, Am. Concrete Inst., Vol. 22, pp. 79–106 (1926). Discussion, pp. 107–109. Data are derived from Table I, p. 83.

TABLE VI.—INDICES OF CONCRETE CONTROL ON JOBS OF AT LEAST FOUR-MONTHS DURATION.
Coefficient of Variation in 28-day Strength of 6 by 12-in. Test Cylinders Moist-Cured.

Item	Job	References and Notes	Approximate Date	Approximate Period Covered by Tests, yr.	Number of Brands and Type of Cement	Number of Test Cylinders	Average Compressive Strength at 28 days, psi.	Coefficient of Variation, corrected for number of tests, per cent	Curing Conditions
No. 1....	Denver Laboratory	1	1940–42	1.7	1 standard	231	4140	5.8	100 per cent humidity, 70 F. temperature.
No. 2....	Maine Experiment Station	2	1936	0.5	3	300±	3370	11.3	Moist sawdust or shavings in field 3 or 4 days, then 97 per cent humidity, 70 F. ± 2 F. in laboratory (p. 9 ref.).
No. 3....	Ohio River Improvement	3	1929	0.7	1	64	4780	11.5	Damp sand at 75 ± 15 F. (p. 1354 ref.).
No. 4....	Chats Falls, Ontario Dam	4	1930–31	1.1	1	1836	3560	11.6	One day in field, then moist room in laboratory (p. 257 ref.).
No. 5....	T.V.A. Dam	5	1935	0.3	3 modified	100	4480	11.7	100 per cent humidity, 70 F. temperature.
No. 6....	Bur. of Recl. dam No. 1	6	1938–40	2.1	10 low heat (blend)	4600	4150	12.5	97½ per cent humidity, 70 F. temperature.
No. 7....	Bur. of Recl. dam No. 4	1	1937–39	1.2	1 low heat	133	4300	12.7	97½ per cent humidity, 70 F. temperature.
No. 8....	Bur. of Recl. dam No. 4	1	1937–41	3.1	1 low heat	503	3645	13.9	97½ per cent humidity, 70 F. temperature.
No. 9....	Univ. Pittsburgh Stadium	7	1924–25	1.2	....	159	2125	14.1	Damp sand, no temperature control (p. 116 ref.).
No. 10...	Los Angeles Storm Drains	8	1928–29	1.0	2	510	3400	14.3	Wet sand 24 hr., then to laboratory (p. 1368 ref.).
No. 11...	Bur. of Recl. Dam No. 4	1	1937–41	3.2	3 low heat	663	3770	15.2	97½ per cent humidity, 70 F. temperature.
No. 12...	Columbus Ohio Sewer, Sec. 1	9	1930	1.0	1	217	3460	15.3	Damp sand or damp room (p. 1365 ref.).
No. 13...	Columbus Ohio Sewer, Sec. 4	9	1929–30	2.0	1	319	3440	16.0	Damp sand or damp room (p. 1365 ref.).
No. 14...	Columbus Ohio Sewer, Sec. 2	9	1930	1.0	1	108	3165	16.6	Damp sand or damp room (p. 1365 ref.).
No. 15...	Bur. of Recl. Dam No. 3	1	1940–41	1.3	1 modified and 4 low heat (blend) 20 per cent pumicite	192	2700	17.5	100 per cent humidity, 70 F. temperature.
No. 16...	Bur. of Recl. Dam No. 2	6	1940–42	2.1	1 low heat	4000	3160	18.2	100 per cent humidity, 70 F. temperature.
No. 17...	Diablo Dam, Washington	10	1928–31	3.3	....	600±	4175	18.6	Wet sand 60–70 F. in field until 2 days before test date, then shipped to laboratory (p. 543 ref.).
No. 18...	Delaware Aqueduct, New York	11	1940–42	2.2	5 moderate heat	501	5045	19	
No. 19...	Bur. of Recl. Dam No. 3	1	1940–41	1.3	3 modified and 4 low heat (blend) 20 and 25% pumicite	255	2560	19.4	100 per cent humidity, 70 F. temperature.
No. 20...	Joint Committee Spec., 1940	12	...	...	....	...	...	20	
No. 21...	Selected Canadian Dams	13	1919–26	...	....	1168	1950	21	
No. 22...	Columbus Ohio Sewer, Sec. 3	9	1928–30	2.0	1	407	3320	21.7	Damp sand or damp room (p. 1365 ref.).
No. 23...	Ohio River Improvement	3	1926	0.7	....	172	2720	23.7	Damp sand, 75 ± 15 F. (p. 1354 ref.).
No. 24...	Wilson Dam, Alabama	14	1923–24	0.9	....	20	2550	24.1	
No. 25...	Ohio River Improvement	3	1928	0.7	5	206	2690	25.8	Damp sand, 75 ± 15 F. (p. 1354 ref.).
No. 26...	Ohio River Improvement	3	1927	0.7	....	236	2685	27.3	Damp sand, 75 ± 15 F. (p. 1354 ref.).
No. 27...	Ohio River Improvement	3	1925	0.7	....	81	2155	38.3	Damp sand, 75 ± 15 F. (p. 1354 ref.)
Average, excluding items Nos. 1 and 20....				1.5			3335	18.1	

References and Notes for Table VI

1. Unpublished data, Denver office, U. S. Bureau of Reclamation. (Test data for item 1 corrected for changes in water-cement ratio.)
2. H. Walter Leavitt, "Predictive Values and Variability of Field Concrete Cylinders from Maine Gravel Aggregates," *Bulletin No. 33*, Maine Technology Experiment Station, University Maine, Orono, Me., May, 1937, 20 pp. Data are taken from Table 3, p. 11.
3. William M. Hall, "Manufacturing Concrete of Uniform Quality," *Transactions*, Am. Soc. Civil Engrs., Vol. 96, pp. 1336–1358 (1932). Discussion pp. 1359–1379. Data are derived from Table 1, p. 1341. Note.—1-in. maximum aggregate (p. 1353).
4. H. L. Trotter and Wilfred Schnarr, "Concreting Problems—Chats Falls Development," *Proceedings*, Am. Concrete Inst., Vol. 29, 1933, pp. 249–274. Data are derived from Table 3, p. 253. Field cylinders were 8 by 16 in. (p. 258).
5. Unpublished data.
6. Table II, this paper.
7. Chas. E. Nichols, Discussion of paper on "Control of Concrete Mixtures on University of Pittsburgh Stadium," by W. S. Hindman, *Proceedings*, Am. Concrete Inst., Vol. 22, pp. 110–118 (1926); Discussion, pp. 119–121. Data are derived from curve on p. 119.
8. C. E. Arnold, Discussion of reference 3 above. Data are derived from curve (a) Fig. 4, p. 1369.
9. O. Bonney and D. T. Mitchell, Discussion of reference 3 above. Data are derived from Table 5, p. 1366.
10. H. F. Faulkner and R. R. Hubbard, "Design and Control of Concrete for Diablo Dam," *Proceedings*, Am. Concrete Inst., Vol. 27, pp. 529–545 (1931). Data are derived from a plot of tabulation on p. 545.
11. Editorial Staff. The Delaware Water Supply News, No. 105, December 1, 1942. Published by Board of Water Supply, City of New York. Data are computed from tabulation pp. 482–483.
12. Report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete, submitting "Recommended Practice and Standard Specifications for Concrete and Reinforced Concrete," Am. Soc. Testing Mats., June, 1940. (Issued as a separate publication.) Data are computed from par. 327-SA, p. 113.
13. Roderick B. Young, "Seven Years of Experience With Job Control of the Quality of Concrete," *Proceedings*, Am. Concrete Inst., Vol. 22, pp. 79–106 (1926); Discussion, pp. 107–109. Data are derived from Table 1, p. 83.
14. John W. Hall, "Control of Mixtures and Testing of Wilson Dam Concrete," *Proceedings*, Am. Concrete Inst., Vol. 22, pp. 488-508 (1926), Discussion, pp. 509-512. Data are derived from Table III, p. 494.

extending over a considerable period, say six months or more, and so situated that field laboratories must be used, concrete of more than average uniformity will have a mean variation of from 12 to 15 per cent."

With these percentages raised to rough values of say 15 to 20 per cent to express them in terms of coefficient of variation, and noting that the average for the 25 construction jobs in Table VI is 18.1, it appears that Mr. Young's statement is still valid.

The next question is: How much of the total variance (square of job coefficient of variation) for the range of jobs listed is chargeable to the cement? Unfortunately the reference data do not supply the answer, since variance studies would be required and the authors are not aware of any such studies other than their own, which gave 20 per cent for dam No. 1, for a job coefficient of variation of 12.5 per cent (item 6 of Table VI), and 40 per cent for dam No. 2, for a job coefficient of 18.2 (item 16 of table). As the variance of $(38.3)^2$ or 1470 for the job showing the highest coefficient is over eleven times as large as the variance of $(11.3)^2$ or 128 for the job with the lowest coefficient, it is obvious that the question of contribution by the cement can be discussed only in generalities. One can be reasonably sure that for job coefficients up to say 13 per cent (variance 169), the variations due to cement will not be large, as cements go, yet the cement may be a substantial contributing factor. To illustrate, if we assume low variances of say 50 for water-cement ratio and concrete temperature combined, and another 50 for experimental error and miscellaneous factors, leaving 69 for the cement, the relative contribution of the cement will be 69/169 or 41 per cent. Such a set of circumstances could readily account for the good parallelism reported by Mr. Freeman between the cement strength and concrete strength curves for one of the T.V.A. dams. For job coefficients of variation larger than say 13 per cent, the variations due to cement and the percentage cement contributions could conceivably range from very small to very large values, depending principally on the control of water-cement ratio and test cylinder handling procedure, and to some extent on the average concrete strength (the lower the strength level, the higher the coefficient of variation). The authors are hopeful that more variance studies will be undertaken by concrete technologists to add to the meager data now available.

Up to this point, the authors' closure has been virtually restricted to the material in Parts I and II of the paper, owing to the discussions of the nine contributors being thus confined. The absence of discussion on Part III, dealing with the practical significance of cement strength variations, is somewhat of an enigma. An informal comment by F. E. Schmitt[25] suggests that the test data in Part III and their conclusions were not accorded the prominence warranted by their importance. Be that as it may, the fact remains that the findings from any study of cement acceptance tests or field concrete control tests, based as these tests are on standardized operating procedures, are at best potential indices of limited application. Consequently, from a practical standpoint such findings should be tempered or interpreted in the light of their bearing on the ultimate behavior of the concrete in the particular structure being built or considered. Unless this is done there is danger that the importance of variations in cement, as indicated by routine tests, will be overemphasized for massive structures and for thin-section structures in humid climates, and possibly underemphasized for thin-section structures located in arid regions or otherwise subject to unfavorable conditions for prolonged cement reaction. It should help to keep in mind that, under like conditions of *continued moist curing*, well-made portland cements of various types, brands, and fluctuating individual compositions and finenesses weave their diversified time-strength patterns, yet eventually reach a more or less common strength level.

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[25] Formerly, Editor of *Engineering News-Record*, now on Denver staff of U. S. Bureau of Reclamation.

EFFECT OF AIR-ENTRAPPING PORTLAND CEMENT ON THE RESISTANCE TO FREEZING AND THAWING OF CONCRETE CONTAINING INFERIOR COARSE AGGREGATE

By E. O. Axon,[1] T. F. Willis,[1] and F. V. Reagel[1]

Synopsis

Use of certain coarse aggregates has been found to be the principal cause for the inferior resistance to freezing and thawing of some concrete pavements in Missouri. Entrapment of air was considered a possibility for improving the resistance of such concrete. This was accomplished by use of portland cement which had been ground with vinsol resin.

Resistance to freezing and thawing (durability),[2] flexural strength, and thermal coefficient of concretes made with four coarse aggregates and varying percentages of air were measured. Of the coarse aggregates included, two were limestones and two were cherts. Curing, and subsequent freezing and thawing were conducted according to two procedures on the chert aggregate concrete, but only one was used on the limestone concrete. The effect of freezing and thawing was determined by measuring the changes in both dynamic modulus of elasticity and flexural strength.

The results obtained by the two test procedures are contradictory as to the effect of entrapped air on the durability of concrete containing one of the cherts and indicate little or no benefit in the case of the other. Results with both limestone concretes containing entrapped air show a definite improvement in durability. Reduction in strength accompanying entrapment of air indicates that care should be exercised in the use of this means of improving durability.

For several years, the Missouri State Highway Department has been investigating the cause and cure for the disintegration of portions of some of its concrete pavements. From periodical observations and maintenance records of some thousand miles of pavement in service, it was found that the most severe deterioration, considering area affected and rate of progression, was associated with the use of certain coarse aggregates. This was true regardless of what fine aggregate and cement had been combined with them. Further study of the climatic conditions where the worst deterioration had occurred, together with laboratory tests of concrete subjected to various artificial weathering cycles, indicated that freezing and thawing was the most active weathering agent causing the disintegration.

As a result of these observations several series of tests have been performed in this laboratory in which the resistance of concrete to freezing and thawing has been studied. The initial objective was the development of an accelerated test procedure which would give relative results, with concretes containing different coarse aggregates, comparable to those obtained from pavements in service. The second objective was to use this test procedure in evaluating various methods of ameliorating the effect of the coarse aggregates, use of which had resulted in serious deterioration of concrete pavements.

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[2] The term durability, throughout this paper, is used in a limited sense to mean only resistance to repeated freezing and thawing.

One of the first attempts at accomplishing the second objective was the use of cement ground with a small amount of vinsol resin. Tests made by the New York Highway Department, the Universal Atlas Portland Cement Co., the Portland Cement Assn., and others have shown that use of this material, as well as other air-entrapping agents, is effective in improving the resistance of concrete pavements to surface disintegration caused by freezing and thawing in the presence of calcium chloride used for ice removal. It was thought that some of the properties reportedly imparted to concrete by the use of this agent would tend to ameliorate the detrimental effect of coarse aggregates, such as had caused trouble in Missouri.

TABLE I.—PROPERTIES OF FINE AGGREGATE.

Percentage retained on	
No. 4 sieve	4
No. 8 sieve	20
No. 14 sieve	33
No. 20 sieve	42
No. 28 sieve	55
No. 48 sieve	88
No. 100 sieve	99
Apparent specific gravity	2.565
Absorption, per cent (30 min.)	0.8
Description: Sand consists essentially of angular grains of chert and quartz.	

SCOPE

Four different coarse aggregates were used in the tests, two limestones (aggregates B and D) and two cherts (aggregates A and C). Based on service records of several hundred miles of pavements, concretes containing these aggregates have been rated as excellent, fair, fair, and poor, respectively. Concrete having air contents of approximately 1, 4 and 7 per cent were tested. The normal portland cement used gave the 1 per cent air content; cement ground with 0.1 per cent vinsol resin was blended with the normal cement to give the 4 and 7 per cent air contents.

Concrete prisms $3\frac{1}{2}$ by $4\frac{1}{2}$ by 16 in. were fabricated three or four to a set. One set for each combination of aggregate, air content, and test procedure was tested in flexure at 35 days, and three or more sets were subjected to freezing and thawing. All combinations of aggregate and air content were subjected to a procedure developed and used extensively in this laboratory. Duplicate specimens of five of the combinations were subjected to a procedure essentially similar to one used by the Portland Cement Assn.

TABLE II.—PROPERTIES OF COARSE AGGREGATE.

	Aggregate A (Chert)	Aggregate B (Limestone)	Aggregate C (Chert)	Aggregate D (Limestone)
Percentage retained on:				
1¼-in. sieve	1	0	0	0
1-in. sieve	12	12	13	12
¾-in. sieve	48	43	42	41
½-in. sieve	73	72	72	72
⅜-in. sieve	85	88	88	88
No. 4 sieve	100	99	100	100
Bulk specific gravity, saturated surface dry	2.526	2.667	2.505	2.650
Absorption, per cent	3.12	1.42	3.73	1.81

MATERIALS USED

Fine Aggregate:

The fine aggregate used in these tests was a river sand having the physical properties shown in Table I. This sand was in an air-dry condition when used.

Coarse Aggregates:

Of the four coarse aggregates used, *B* and *D* are limestones, and *A* and *C* are cherts. The coarse aggregates were used in a saturated condition. The limestones were saturated under vacuum; the cherts were "stream-wet." The physical properties of these aggregates are shown in Table II.

Cement:

The normal portland cement and the portland cement containing vinsol resin were obtained by thoroughly grinding blended clinker to approximately the same fineness in a laboratory mill. One tenth per cent of vinsol resin, by weight, was ground with the clinker in making the portland cement containing vinsol resin. By combining these two cements in various percentages, the air content of the concrete could be varied as desired. The physical properties of the cements are shown in Table III.

TABLE III.—PHYSICAL PROPERTIES OF CEMENT.

	Type of Cement	
	Normal	0.1 per cent Vinsol Resin Added
Normal consistency, per cent	23.0	23.0
Soundness	OK	OK
Initial set	3 hr. 35 min.	3 hr. 35 min.
Final set	5 hr. 35 min.	5 hr. 35 min.
Tensile strength, psi.		
7 days	330	250
28 days	432	307
Specific surface, turbidimeter	1787	1757
Percentage passing No. 325 sieve	83.23	83.05
Autoclave expansion, per cent	+0.23	+0.24
Specific gravity	3.01	2.95
Chloroform soluble, per cent	0.01	0.09

CONCRETE PROPORTIONS

The mixes for this series of tests were designed so that the cement factor, the consistency, and the absolute volume of coarse aggregate per unit volume of concrete were kept as nearly constant as possible. The air content was varied intentionally, and in order to maintain a constant consistency it was necessary to vary the water-cement ratio. Thus to maintain a constant cement factor it was necessary to vary the sand content of the mixes to compensate for the variations in air content and water-cement ratio. Since it was impossible to predict accurately the air content of any given combination of materials, or the quantity of water required for constant consistency, minor differences from the designed quantities of all materials occurred in the mixtures as used.

TABLE IV.—PROPORTIONS AND CONSISTENCY OF PLASTIC CONCRETE.[a]

Coarse Aggregate	Approximate Air, per cent	Vinsol Resin,[b] per cent	Actual Air, per cent	Proportions by Absolute Volume	Water-Cement Ratio	Cement Factor, bbl. per yd.	Slump, in.
A	1	0	0.85	1:2.78:3.99	0.730	1.438	3.00
	4	0.017	4.25	1:2.54:3.97	0.689	1.438	3.60
	7	0.034	7.35	1:2.33:3.96	0.647	1.436	3.20
B	1	0	0.55	1:2.70:3.99	0.765	1.444	3.00
	4	0.019	4.20	1:2.48:3.97	0.721	1.439	3.25
	7	0.034	7.15	1:2.27:3.96	0.684	1.437	3.45
C	1	0	0.80	1:2.76:3.98	0.747	1.437	3.00
	4	0.0175	4.25	1:2.54:3.97	0.694	1.436	3.15
	7	0.034	7.20	1:2.33:3.96	0.659	1.436	3.10
D	1	0	0.35	1:2.56:3.99	0.795	1.443	3.25
	4	0.0193	4.00	1:2.48:3.96	0.731	1.438	3.25
	7	0.034	6.90	1:2.20:3.96	0.702	1.435	3.10

[a] Average of at least three batches of concrete.

[b] Calculated from proportions of portland cement containing vinsol resin and normal portland cement used.

The basic concrete mixture used in these tests with normal cement was similar to that used in most of the pavements in Missouri constructed some 11 to 17 yr. ago. It is in some of these pavements that the worst deterioration has occurred. The mixture approximates 1.00:2.10:3.45 by dry rodded volumes, with a cement factor of 1.45 bbl. per cu. yd., and a water-cement ratio of 0.75. During the era used, and with most aggregates, the mix was considered to be oversanded, highly workable at a $2\frac{1}{2}$ to $3\frac{1}{2}$-in. slump without serious tendency to bleed, and in general an excellent pavement mixture.

Data pertaining to the proportions are shown in Table IV.

MIXING

All materials were batched by weight and the concrete was mixed in a model SW Lancaster mixer. Each batch was mixed 30 sec. dry and 2 min. wet. The size of the batch varied from 0.325 to 1.625 cu. ft., depending on the number of beams to be made for each combination of materials.

RESULTS OF OBSERVATIONS AND TESTS ON THE PLASTIC CONCRETE

As soon as possible after the mixing was completed a slump test and a determination of the air content of the concrete were made simultaneously. The results obtained by these tests are shown in Table IV.

From these results it can be seen that the air contents varied slightly from those intended and that the slumps obtained were not absolutely uniform. If these variations have any significant effect on the results to be described, it is not apparent from the data. Observation of the various mixtures containing vinsol resin showed them invariably to be "fatter" and more "placeable," to have less tendency toward segregation, and to have reduced tendency to bleed.

FABRICATION OF TEST SPECIMENS

Metal beam molds $3\frac{1}{2}$-in. wide by $4\frac{1}{2}$-in. deep by 16-in. long were used. While no attempt was made to waterproof the joints the molds were reasonably tight. The beams were molded in two layers, and each layer was rodded 50 times with a $\frac{5}{8}$-in. bullet-pointed rod. The sides and ends were spaded, and the surface was struck off and finished with a wooden float. After fabrication all the beams were immediately transferred to the moist room.

At least three specimens were made for each variable and each end point in the tests. Hence each tabulated value or plotted point is an average of the results of at least three, and in most cases, four specimens.

CURING

All beams were cured by one or the other of two methods, designated Missouri and P.C.A. The so-called P.C.A. method, although differing somewhat in minor details, is essentially similar to one used by the Research Laboratory of the Portland Cement Assn. Details of the two methods are given below.

Missouri Method:

Step (*a*)..........	2 days in molds in moist room.
Step (*b*)..........	5 days on shelves in moist room.
Step (*c*)..........	28 days in water.

P.C.A. Method:

Step (*a*)..........	2 days in molds in moist room.
Step (*b*)..........	5 days on shelves in moist room.
Step (*c*)..........	7 days in water.
Step (*d*)..........	14 days air drying in laboratory.
Step (*e*)..........	7 days in water.

The moist room was maintained at a temperature of 70 ± 2 F. and a relative humidity of 95+. The room in which the beams were air dried, under the P.C.A. method, was subject to the normal fluctuations in temperature and humidity of the rest of the laboratory. The approximate temperature variation was from 65 to 80 F., and the average relative humidity was approximately 31.

The two curing methods are identical up to the fourteenth day. From the fourteenth to the twenty-eighth day, beams cured by the P.C.A. method are drying while those cured by the Missouri method continue in a saturated state or under conditions permitting that state to be approached. From the twenty-eighth to the thirty-fifth day the methods are similar, all beams being in water.

The effects of these two different curing methods on the moisture content of the beams at the end of the curing period are pointed out later.

TESTS ON HARDENED CONCRETE

Changes in Moisture Content as Reflected by Changes in Weight:

All beams were weighed periodically during the curing period, the initial weight being obtained when beams were 2 days old. Since care was taken to prevent, and there was no evidence of, mortar loss, it was assumed that the changes in weight were due to changes in moisture content. Average changes in weight are shown in Table V.

TABLE V.—PERCENTAGE CHANGE IN WEIGHT DURING CURING PERIOD, BASED ON WEIGHT AT 2 DAYS AS 100 PER CENT.

Age, days	Aggregate A (Chert), Absorption 3.12 per cent			Aggregate B (Limestone), Absorption 1.42 per cent			Aggregate C (Chert), Absorption 3.73 per cent			Aggregate D (Limestone), Absorption 1.81 per cent	
	0.85 Per cent Air in Fresh Concrete	4.25 Per cent Air in Fresh Concrete	7.35 Per cent Air in Fresh Concrete	0.55 Per cent Air in Fresh Concrete	4.20 Per cent Air in Fresh Concrete	7.15 Per cent Air in Fresh Concrete	0.80 Per cent Air in Fresh Concrete	4.25 Per cent Air in Fresh Concrete	7.20 Per cent Air in Fresh Concrete	0.35 Per cent Air in Fresh Concrete	4.00 Per cent Air in Fresh Concrete
MISSOURI CURING METHOD											
7........	+0.03	+0.02	+0.01	+0.11	+0.04	+0.08	+0.02	+0.02	+0.04	+0.12	+0.09
14........	+0.18	+0.23	+0.25	+0.32	+0.26	+0.38	+0.21	+0.23	+0.27	+0.36	+0.32
28........	+0.28	+0.38	+0.44	+0.42	+0.41	+0.59	+0.31	+0.39	+0.45	+0.45	+0.48
35........	+0.32	+0.43	+0.49	+0.45	+0.46	+0.65	+0.34	+0.43	+0.51	+0.49	+0.53
P.C.A. CURING METHOD											
7........	+0.01	0.00	−0.02	+0.09	...	...	+0.01	+0.03	+0.02	...	...
14........	+0.20	+0.25	+0.23	+0.34	...	...	+0.23	+0.23	+0.26	...	...
28........	−2.33	−2.48	−2.44	−1.94	...	...	−2.56	−2.62	−2.71	...	...
35........	−0.53	−0.55	−0.49	−0.13	...	...	−0.45	−0.48	−0.48	...	...

These results indicate that, at the end of the curing period, specimens cured by the P.C.A. method were not only drier than specimens cured by the Missouri method but also had a lower moisture content at the end of the curing period than they had at the start of the drying period. Apparently for the conditions existing in this series, a 7-day soaking period is not sufficient to resaturate concrete that has been subjected to a 14-day drying period. Undoubtedly, the use of saturated coarse aggregate influenced the magnitude of the above differences. Also the moisture content of the coarse aggregate affected the quantity of moisture lost from the beams during the air-drying period under the P.C.A. method, this quantity varying directly with the absorption of the coarse aggregate.

Under the P.C.A. curing procedure the use of portland cement containing vinsol resin had very little, if any, effect on the rate of loss or gain in moisture during the curing period. However, under the Missouri curing procedure the concretes with 4 and 7 per cent air absorbed water at a somewhat greater rate than comparable concretes with normal cement. While the rate of absorption was greater, concretes with 4 and 7 per cent air actually contained less total water when freezing was started due to difference in initial water-cement ratio, than comparable concretes made from normal cement. Thus, regardless of the curing method, the above facts together with the difference in the original air content of the concretes, indicates that the degree of saturation of concrete with 4 and 7 per cent air was considerably less when freezing started than that of comparable concretes with normal cement.

The differences in the moisture content of specimens at the time of initial freezing is pointed out because of the known effect of contained moisture on the rate of deterioration of concrete under freezing and thawing. Presently there is too little information to judge which of the two curing methods, combined with some freezing-and-thawing procedure, will give results most comparable to those obtained in service.

TABLE VI.—DYNAMIC MODULUS OF BEAMS DURING CURING PERIOD AND FLEXURAL STRENGTH OF CONTROL BEAMS.

Age, days	Aggregate A			Aggregate B			Aggregate C			Aggregate D		
	0.85 Per cent Air in Fresh Concrete	4.25 Per cent Air in Fresh Concrete	7.35 Per cent Air in Fresh Concrete	0.55 Per cent Air in Fresh Concrete	4.20 Per cent Air in Fresh Concrete	7.15 Per cent Air in Fresh Concrete	0.80 Per cent Air in Fresh Concrete	4.25 Per cent Air in Fresh Concrete	7.20 Per cent Air in Fresh Concrete	0.35 Per cent Air in Fresh Concrete	4.00 Per cent Air in Fresh Concrete	6.90 Per cent Air in Fresh Concrete
DYNAMIC MODULUS, PSI. × 10^{-6}—MISSOURI CURING METHOD												
7	5.1	4.7	4.3	4.7	4.4	4.0	5.1	4.7	4.3	4.6	4.4	...
14	5.3	4.9	4.6	5.1	4.8	4.4	5.3	4.9	4.5	4.9	4.7	...
28	5.6	5.1	4.7	5.35	5.0	4.6	5.5	5.1	4.7	5.1	4.8	...
35	5.6	5.2	4.8	5.45	5.1	4.7	5.6	5.1	4.8	5.2	4.9	...
DYNAMIC MODULUS, PSI. × 10^{-6}—P.C.A. CURING METHOD												
7	5.1	4.7	4.3	4.7	...	...	5.1	4.6	4.4	...	...	...
14	5.3	4.9	4.5	5.1	...	...	5.3	4.9	4.5	...	...	...
28	4.9	4.4	4.1	4.8	...	...	4.8	4.4	4.0	...	...	...
35	5.35	4.9	4.55	5.2	...	...	5.3	4.9	4.6	...	...	...
FLEXURAL STRENGTH, PSI.—MISSOURI CURING METHOD[a]												
35	835	805	710	945	865	775	780	725	655	890	820	675
FLEXURAL STRENGTH, PSI.—P.C.A. CURING METHOD[a]												
35	770	725	675	880	...	685	730	660	590	...	...	610

[a] Average of 3, 4 or 7 beams. Depth 3½ in., span 14 in., midpoint loading.

Dynamic Modulus and Flexural Strength During Curing Period:

The dynamic modulus of all beams was measured at intervals during the curing period, and sets of control specimens were tested for flexural strength at the end of the curing period. The results obtained are shown in Table VI.

These results show that both the dynamic modulus and the flexural strength are affected by the kind of coarse aggregate used, the air content of the concrete, and the method of curing used. For any particular combination of materials, age, and curing procedure, both these properties vary inversely with the air content. With most combinations the proportionate effect on the flexural strength is much greater than it is on the modulus of elasticity. The magnitudes of both of these properties, at the end of the curing period, were greater for specimens cured by the Missouri procedure than for comparable specimens cured by the P.C.A. procedure.

Evidence that the dynamic modulus varies with the moisture content of the specimens is shown by the fact that the dynamic modulus increases during water curing and decreases during air curing.

Temperature Coefficient of Expansion:

For specimens cured by the Missouri procedure, measurements of the length changes when the temperature was reduced from 70 F. to 40 F. at the end of the curing period were used to calculate the temperature coefficient of expansion. For the particular conditions existing at the time of testing, the results show that the type of coarse aggregate was the controlling factor in determining the temperature coefficient of expansion; and that any effect of vinsol resin was somewhat less than 0.000001 in. per inch per deg. Fahr.

The temperature coefficients for concretes made with the four coarse aggregates were 0.000006 for cherts *A* and *C*, 0.000003 for limestone *B*, and 0.000004 for limestone *D*.

TEST PROCEDURES FOR FREEZING AND THAWING SPECIMENS

All concretes—combinations of the four aggregates and three air contents—were subjected to the Missouri freezing-and-thawing procedure. Due to the limited facilities available, only five of the combinations were subjected to the P.C.A. procedure.

Under the Missouri procedure, specimens were frozen by placing in the air of a cold room, the temperature of which was maintained at 0 F., for 10 hr., and were thawed by placing in water at a temperature of 40 F. for 2 hr. A complete cycle was 12 hr. and specimens were subjected to two cycles daily until completion of the test.

Under the P.C.A. procedure, the specimens were frozen in water in rubber containers slightly larger than the specimens. Containers were placed in a brine solution that was maintained at −10 to −20 F. The freezing period varied slightly, being of sufficient duration to lower the temperature at the center of the specimens to approximately 5 F., but averaged 55 min. Specimens were thawed by removing the containers from the brine and placing in a tank containing water at 80 F., which was decreased to 60 F. in 1 hr. and maintained at this temperature. The thawing period varied slightly, but averaged 3 hr. and 5 min. A complete cycle required 4 hr. Specimens were subjected to six cycles daily until the tests were completed.

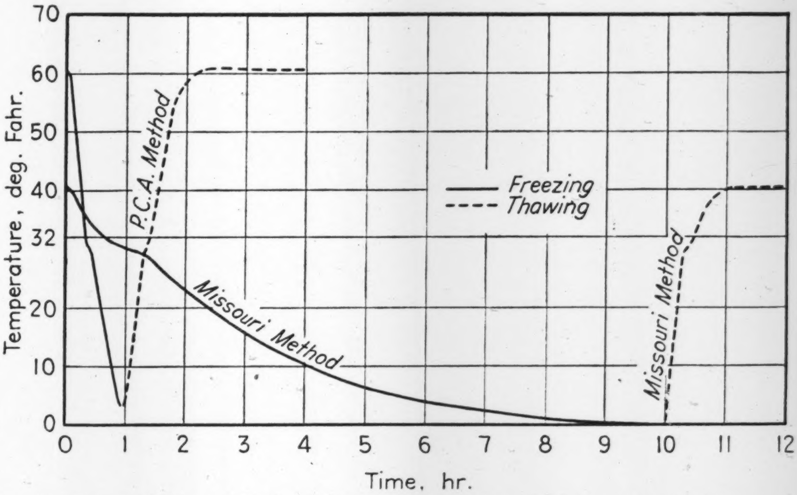

FIG. 1.—Internal Concrete Temperatures Typical of the Two Freezing-and-Thawing Cycles Used.

Internal concrete temperatures typical of the two freezing-and-thawing cycles used are shown in Fig. 1.

DISCUSSION OF RESULTS OF FREEZING-AND-THAWING TESTS

A complete discussion of the relative merits of various test procedures, and methods of treatment of the data obtained with them, is beyond the scope of this paper. However, in amplification of the interpretation of the data of these tests, some exposition is desirable.

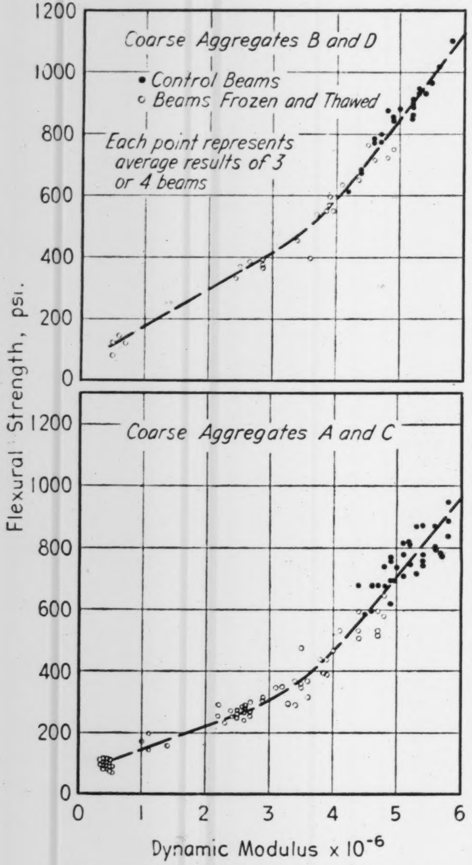

FIG. 2.—Relation of Flexural Strength to Dynamic Modulus.

Detailed information on the relative effect of many of the factors involved in the freezing and thawing of concrete structures in service is very meager. Under this condition there can be no assurance that an arbitrarily selected laboratory test procedure will reflect the disintegration that would occur under natural conditions. In this laboratory the attempt has been made to find a test procedure which, when applied to various concretes similar in composition to those in pavements of known service record, would qualitatively reflect the same relative resistance to freezing and thawing as was indicated by the service records of pavements containing these concretes. When used for the purpose for which it was developed, namely, the effect of different coarse aggregates on the relative durability of the concretes containing them, the procedure has thus far been satisfactory. Until this series of tests, the procedure had been applied only to the comparison of concretes of approximately similar proportions and water-cement ratio—the only variable being the type and source of the coarse aggregate.

In this series of tests, however, an attempt was made to evaluate the effect of an air-entrapping portland cement on the durability of concretes containing different aggregates. Use of this admixture changes some of the properties of the cement paste and, as previously pointed out, the water-cement ratio was varied to obtain a constant consistency. The test has not been previously used to evaluate effect of variations in the characteristics of mortars, and no field service records are available with which to compare results. Hence, the authors are not dogmatic about the value of the Missouri test procedure or the results indicated by it.

This was one of the reasons that duplicate specimens of some of the combinations were subjected to the test procedure of the Research Laboratory of the Portland Cement Assn. This procedure, one of several employed by that laboratory, has been used to test the effect of air-entrapping portland cement on the progressive mortar disintegration in pavements caused by the freezing and thawing accompanying applications of calcium chloride for ice removal.

As with the conditions surrounding the freezing and thawing of concrete in service, little is known of the mechanics of the resistance of concrete to the disintegrating forces of freezing and thawing. Measurement of the deterioration of concrete under freezing and thawing is attempted in this laboratory by determining the dynamic modulus of elasticity of concrete prisms before and subsequent to freezing and thawing. This property of the concrete can be measured without destroying the test specimen and, as pointed out by several investigators, is related to the flexural strength. Thus an approximation of the change in strength can be obtained without testing the large number of specimens which would be necessary in order to measure the flexural strength directly.

The customary method for illustrating and comparing the results of freezing-and-thawing tests of concretes is a plot of percentage loss in dynamic modulus *versus* number of cycles of freezing and thawing. This device, one of several studied, was adopted because the relative position and shape of the curves, for a number of tests on concretes similar in composition to those used in pavements of known service record, was about what would have been expected from those records, and because of the simplicity of the necessary calculations. In all freezing-and-thawing investigations prior to this series, prime interest was in the relative resistance of concretes having similar mortar characteristics and approximately equal strengths. Because of this, little attention was paid to the actual magnitude of either the initial strength or the ultimate strength at whatever end point was chosen. It is obvious that the concrete in any structure has functions other than resistance to freezing and thawing to fulfill. For each type of structure there is some minimum strength required to prevent failure from stress other than that caused by freezing and thawing. Hence for evaluating concretes which have no established service record, or concretes which basically are considerably different in initial strength, the actual magnitudes of the strengths at the end point of the freezing-and-thawing test, or the number of cycles required to reach an assumed critical strength, as well as the rates of deterioration should be considered.

For this series of tests no service records of concretes made with portland cement containing vinsol resin were available, and furthermore the initial and ultimate strengths of these concretes differed considerably from those of the concretes with which comparisons are made. On the other hand, the end point for the laboratory freezing-and-thawing test has not been established, the critical strength for pavement concrete is not known, and the percentage loss in dynamic modulus is now widely used as a criterion of durability. For these reasons, the relative deterioration of the concretes are shown in graphs plotted in the customary manner and also by plotting flexural strengths, calculated by a device to be explained later, against cycles of freezing and thawing.

The indications of the freezing-and-thawing tests on concretes designed to contain 7 per cent air were, in all cases studied, similar in trend and magnitude of effect to those of the comparable concretes containing 4 per cent air. The small differences shown are not considered significant, and due to the effect of the entrapment of air on flexural strength, it is believed that concretes containing 4 per cent air would be more generally used than concrete containing

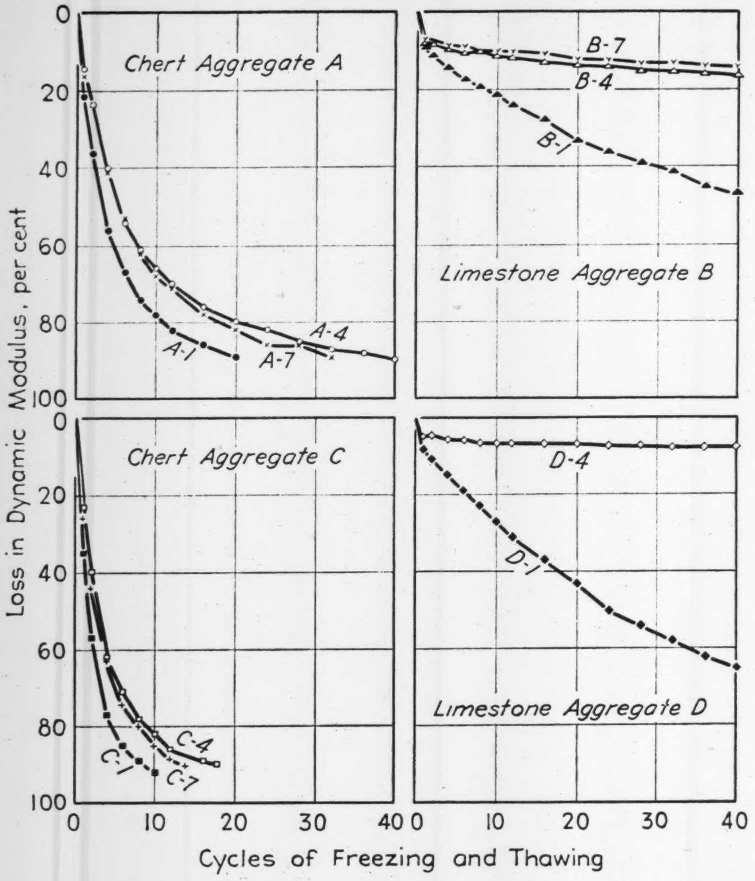

FIG. 3.—Effect of Air-Entrapping Portland Cement on Resistance of Concrete to Freezing and Thawing as Measured by Percentage Loss in Dynamic Modulus. Missouri procedure.

The letters designate the coarse aggregate used, and the numbers indicate the approximate air content of the concrete.

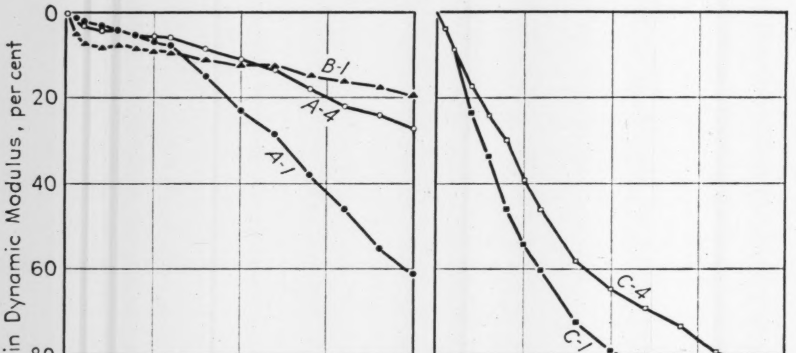

FIG. 4.—Effect of Air-Entrapping Portland Cement on Resistance of Concrete to Freezing and Thawing as Measured by Percentage Loss in Dynamic Modulus. P.C.A. procedure.

The letters designate the coarse aggregate used, and the numbers indicate the approximate air content of the concrete.

7 per cent air. Thus in the following discussion, while the comment pertaining to vinsol resin concrete is generally applicable to concretes of both 4 and 7 per cent air content, it is more specifically applied to and derived from the concretes containing 4 per cent air.

Results are plotted in Figs. 3, 4, 5, and 6. The various curves are identified by combinations of a letter and a number. The letter designates the coarse aggregate used, and the number indicates the approximate air content of the concrete.

In Figs. 3 and 4 the percentage loss in dynamic modulus *versus* cycles of freezing and thawing is shown for the two test procedures. Figure 3 contains the data obtained by the Missouri test procedure. It is indicated that for the highly absorptive chert aggregates A and C the use of air-entrapping portland cement slightly improved the resistance of the concrete to freezing and thawing under the conditions of these tests. However, it is doubtful that this indicated improvement is beyond the range of experimental error, and hence it is not considered to be significant. With neither aggregate is the improvement sufficient to indicate that concrete containing the air-entrapping portland cement would be in the same class with that containing normal portland cement and limestone aggregate B; this being the concrete that had a satisfactory service record in Missouri. With the limestone aggregates B and D, however, the use of the air-entrapping portland cement is indicated to have caused a considerable improvement in the resistance of the concrete to freezing and thawing, greater improvement being shown for aggregate D than for aggregate B. The concrete containing 4 per cent of air and either of these aggregates showed very little deterioration after a small drop during the first two cycles. Both are superior to the corresponding concretes made with the normal cement.

In Fig. 4 the results obtained by the P.C.A. test procedure are plotted in a manner similar to that used in plotting Fig. 3, that is, percentage loss in dynamic modulus *versus* number of cycles of freezing and thawing. Results of some of the concretes shown in Fig. 3 do not appear in Fig. 4, because of the limited facilities available in this laboratory for duplicating the P.C.A. test procedure. Of the aggregate-air content combinations tested, the concrete containing aggregate B and normal cement showed the greatest resistance to freezing and thawing. Concrete containing chert aggregate A and air-entrapping portland cement is indicated to be superior to concrete containing the same aggregate and normal portland cement and practically on a par with concrete containing limestone aggregate B and normal cement. This will be noted as decidedly different from the results obtained by the Missouri test procedure and plotted in Fig. 3. With chert aggregate C the use of air-entrapping portland cement is indicated to have improved the concrete somewhat over that made from the same aggregate and normal cement. However, the improvement is only slight and probably not sufficient to be of any value in pavement concrete.

Figures 5 and 6 show the test results obtained by the two test procedures plotted in a different manner. The flexural strength of the concrete is plotted as an ordinate and the cycles of freezing and thawing as abscissa. The flexural strengths used in plotting these curves were not actually measured for each of the coordinate points but were obtained by interpolation from curves in which measured flexural strength was plotted against dynamic modulus (Fig. 2). The encircled points on the diagrams, however, are the average results for actual flexural strengths of three or more beams tested at the average number of cycles indicated. The proximity of these points to the curves indicates that the

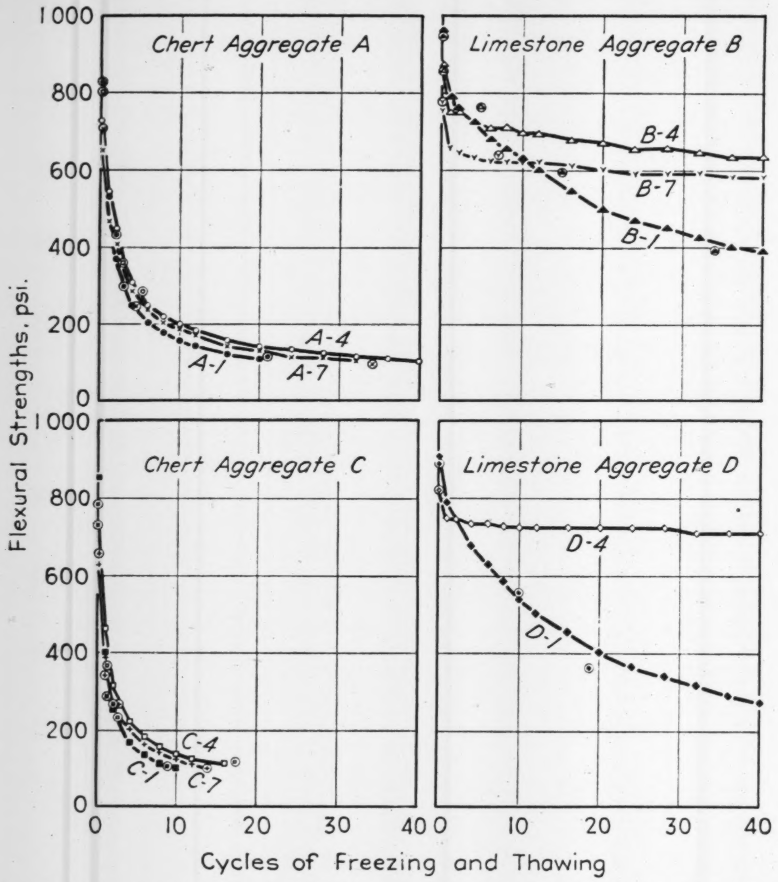

FIG. 5.—Effect of Air-Entrapping Portland Cement on Resistance of Concrete to Freezing and Thawing as Measured by Changes in Flexural Strengths. Missouri procedure.

The letters designate the coarse aggregate used, and the numbers indicate the approximate air content of the concrete.

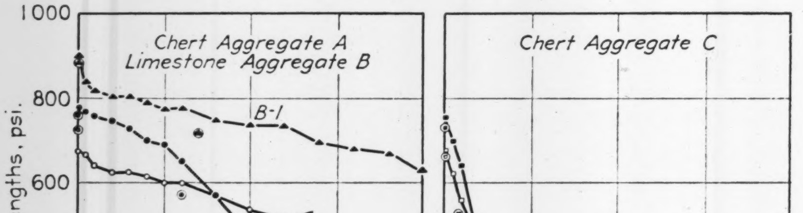

FIG. 6.—Effect of Air-Entrapping Portland Cement on Resistance of Concrete to Freezing and Thawing as Measured by Changes in Flexural Strengths. P.C.A. procedure.

The letters designate the coarse aggregate used, and the numbers indicate the approximate air content of the concrete.

interpolated values of the flexural strengths are reasonably good.

The results obtained by the Missouri method are shown in Fig. 5. By comparison with Fig. 3, it is obvious that the indications of the curves of the two figures are, at least qualitatively, similar. Thus the interpretation of the indications of the results using the Missouri test procedure would be the same, regardless of whether percentage loss in dynamic modulus, or flexural strength is used as a criterion.

Comparison of Fig. 6, showing the results obtained by the P.C.A. test procedure, with Fig. 4 shows similar results for aggregate C, both with respect to the effect of the use of vinsol resin and the relative resistance of this concrete to freezing and thawing as compared with aggregate B. However, the indications of the results with aggregate A are somewhat different from those exhibited by Fig. 4. Although the concrete containing 4 per cent air (A-4) has a somewhat greater resistance than the concrete with the same aggregate and normal cement (A-1), as indicated by the relative slopes and positions of the two curves, the difference is not so great as is indicated in Fig. 4. Moreover, the A-4 curve in Fig. 6 lies considerably below the B-1 curve, and the flexural strength indicated by it, after comparatively few cycles of freezing and thawing, is very possibly less than the critical strength for pavement concrete. If this be true, it is doubtful that concrete A-4, in the proportions used in these tests, would be much better than the same concrete with normal cement; and it is probable that it would be inferior to B-1, the concrete having an excellent service record.

TABLE VII.—SUMMARY OF INDICATIONS OF TEST RESULTS AS TO EFFECT OF AIR-ENTRAPPING PORTLAND CEMENT ON DURABILITY OF CONCRETES MADE WITH THE VARIOUS AGGREGATES.

	Percentage Loss in Dynamic Modulus		Calculated Flexural Strength	
	Missouri	P.C.A.	Missouri	P.C.A.
Chert aggregate A Service record "Fair"	Not beneficial	Definitely beneficial	Not beneficial	Possibly beneficial but doubtful
Limestone aggregate B Service record "Excellent"	Definitely beneficial		Definitely beneficial in spite of somewhat lower initial strength	
Chert aggregate C Service record "Poor"	Not beneficial	Not beneficial	Not beneficial	Not beneficial
Limestone aggregate D Service record "Fair"	Definitely beneficial		Definitely beneficial in spite of somewhat lower initial strength	

As indicated by Fig. 6, the rate of deterioration under freezing and thawing is about the same for concrete A-4 and concrete B-1, but the initial flexural strength of the former is about 200 psi. less. There is a possibility that if sufficient cement were added to bring the initial flexural strength of concrete A-4 up to that of concrete B-1, the apparent resistance to freezing and thawing, at least as indicated by the P.C.A. test procedure, would be approximately the same.

In order to facilitate the visualization of the significance of the preceding discussion, a summary of the indications of the results obtained by the two test procedures using both percentage loss in dynamic modulus and calculated flexural strength as criteria of durability is presented in Table VII.

There is a reasonable concordance in the indications listed regardless of test procedure or criterion of durability used except in the case of aggregate A. The cause for the discrepancy between the results obtained by the two test procedures in this case is not known.

There is a radical difference in the effect of entrapped air on the results obtained with the two different mineralogical types of aggregate. Obviously the use of vinsol resin produced little or no benefit regardless of test procedure or method of evaluation of durability in the case of one chert aggregate (C), and with the other chert aggregate (A) the benefit is doubtful. On the other hand, as indicated by the one procedure used (Missouri) there is a decided benefit produced by the use of vinsol resin with each of the two limestone aggregates. This does not warrant the indiscriminate use of entrapped air with concrete containing limestone aggregate. For instance, concrete pavements containing aggregate B and normal cement have evidenced little lack of durability. Failures do occur, particularly in the older pavements, but are practically all structural. Under such conditions use of air-entrapping portland cement should be approached with caution. There can be no logic in improving the durability of a concrete at the expense of its structural strength when the inherent characteristics of the concrete are such that failures occurring are the result of inability to resist structural stresses and not those resulting from weathering.

CONCLUSIONS

The use of vinsol resin ground with the clinker to produce an air-entrapping portland cement has certain definite effects on concrete. For concrete in the plastic state, it makes the mixture "fatter," improves placeability, and reduces bleeding. For concrete in the hardened condition it reduces the flexural strength and dynamic modulus of elasticity, at least up to 35 days; but it apparently has little, if any, effect on the thermal coefficient of expansion.

In the present stage of development of laboratory freezing-and-thawing tests the information obtained from these tests can only be considered as indications. These are summarized as follows:

1. Vinsol resin when ground with clinker to produce an air-entrapping portland cement caused no appreciable improvement in durability of concrete containing chert aggregate of such characteristics that its use in pavement concrete has resulted in pavements of poor durability.

2. In the case of chert aggregate (A) of such characteristics that its use in concrete has resulted in a fair service record, there is disagreement between the indications of the two test procedures which raises some question as to the validity of the results of either or both procedures.

In the opinion of the authors there is only a slight chance that an air-entrapping portland cement will appreciably reduce the rate of disintegration resulting from freezing and thawing of pavement concrete containing aggregate of this kind. However, further investigation should be made of this and also the possibility that by increasing the initial flexural strength to balance the loss in strength due to entrappment of air, the durability may be improved.

3. The entrappment of air improved the durability of concretes containing either limestone.

4. For combinations of materials for which tests indicate air-entrapping portland cement will improve the durability, the effect of the entrapped air on the strength of the concrete should be considered and balanced against the need for improved durability.

DISCUSSION

Mr. Charles E. Wuerpel.[1]—This paper is of considerable interest to us since we have found very similar indications in studies which were made for the St. Lawrence Waterway Project in which chert gravel was not used, but a very inferior gravel was compared with aggregate of good quality.

We have an exposure station at Treat Island, Maine, in the most eastern and northern part of the United States, which we consider to provide the most rapidly accelerated natural freezing and thawing which can be obtained on this continent, where extensive investigations were made of the aggregates which would be available for the construction of structures in the St. Lawrence Waterway Project. Plain cements and vinsol resin treated cements were investigated with these aggregates and it was found that the durability of the vinsol resin treated cement specimens containing the good aggregates was very materially better than the plain cement specimens containing similar aggregate, but there was a relatively insignificant improvement in the durability of the concrete with the poor aggregates. The work done by the authors corroborates the conclusion we have reached that vinsol resin cannot be depended upon to protect inferior aggregate. Perhaps I express it differently than the authors did, but, even though you do reduce the water-cement ratio sufficiently to bring the flexural strength into equality with plain cement concrete, you still do not get an appreciable increase in the durability of the concrete containing inferior aggregate.

Mr. A. D. Conrow.[2]—I have had an opportunity to conduct some tests similar to those described in this paper, though under somewhat different conditions. We have in our part of the country (Kansas and Nebraska) quite large quantities of rather fine aggregate. We call it sand-gravel. Sometimes under certain conditions we wish to incorporate a portion of coarse aggregate, varying in quantity from 15 to 40 per cent. We call this "sweetening with coarse aggregate."

Recently I had an opportunity to conduct some tests on such concretes, incorporating an unsound coarse aggregate with the sand-gravel in quantities over a range, I will say, of 10 to 50 per cent. The series of concretes tested thus contained variable amounts of the unsound aggregate.

I found considerable protection from the resin-treated cement, up to a quantity of 25 per cent unsound coarse aggregate. Concrete containing unsound aggregate in quantities greater than this was weakened quite rapidly.

The extent of these tests was not sufficient that I would wish to draw definite conclusions from them, but I offer the information as one experience.

This paper brings out some information which I have desired for some time. That is, the relationship between the dynamic modulus indications and the modulus of rupture. We have facilities in our laboratory for determining the dynamic modulus, but have not until recently had testing equipment to test for modulus of rupture. I have been wishing for some information as to whether the dynamic modulus would give indication of strength changes similar to that which would be shown by modulus of rupture. These tests would indicate there is fairly good correlation. Such a condition may make it possible for us to rely on the dynamic modulus as a measure of damage caused by adverse exposure. At least, the results shown in these tests will give us more confidence in the indications of the dynamic modulus test.

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[2] Research Engineer, Ash Grove Lime & Portland Cement Co., Chanute, Kans.

There is one other thing I might mention in connection with this particular series of tests. Apparently the resin-treated cement used in these tests was a laboratory prepared product. We have had little success in our laboratory in preparing a laboratory product treated with resin that performs the same as the product from mill manufacture. However, if we may assume that the improvement in durability is a result of air entrainment, it is probable it does not matter whether the cement was similar to mill manufacture or not, provided we have the air entrainment as is shown. So it is quite possible that these results on a laboratory prepared cement may reflect what would be obtained had a mill produced product been used.

MR. STANTON WALKER.[3]—This paper contains a great deal of interest to members of the sand and gravel industry. Gravels of chert origin are produced by the industry in large quantities in Missouri, Tennessee, Kentucky, and other states principally to the south of those named. The National Sand and Gravel Assn. has conducted, and is conducting, investigations with the same objective as that described, namely, improving the resistance of chert-gravel concrete to freezing and thawing. This work is being carried out by the Association's Research Foundation at the University of Maryland.

A limited number of exploratory tests have been made with cements containing grinding aids (other than vinsol resin), with a "waterproofing" cement, and with certain admixtures. While some increased resistance was evidenced, the results are not promising of a solution to the main problem. Most promise is offered by investigations involving the moisture condition of the concrete.

It should be emphasized that the problem represented by chert as a concrete aggregate differs depending on circumstances. Cherts vary greatly in physical characteristics. They range from hard, dense, flint-like particles to lightweight porous particles. Some chert gravels have good service records; others have spotty service records, ranging from bad to good. Concrete disintegration that has been attributed to chert gravel has occurred, for the most part, in road slabs. No difficulties of moment have been encountered in structures. It is clear that the severity of climatic conditions is of considerable importance. Such disintegration as has occurred is predominantly in the northern portion of the chert gravel area. No questions of importance with respect to durability have arisen in the southern portion of the area.

Under the more severe climatic conditions it is evident that the quality of chert gravel and the environment of use of any of the chert gravels are important factors. Any chert gravel concrete road showing a high proportion of disintegration also shows numerous sections in

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[3] Director of Engineering, National Sand and Gravel Assn., Washington, D. C.

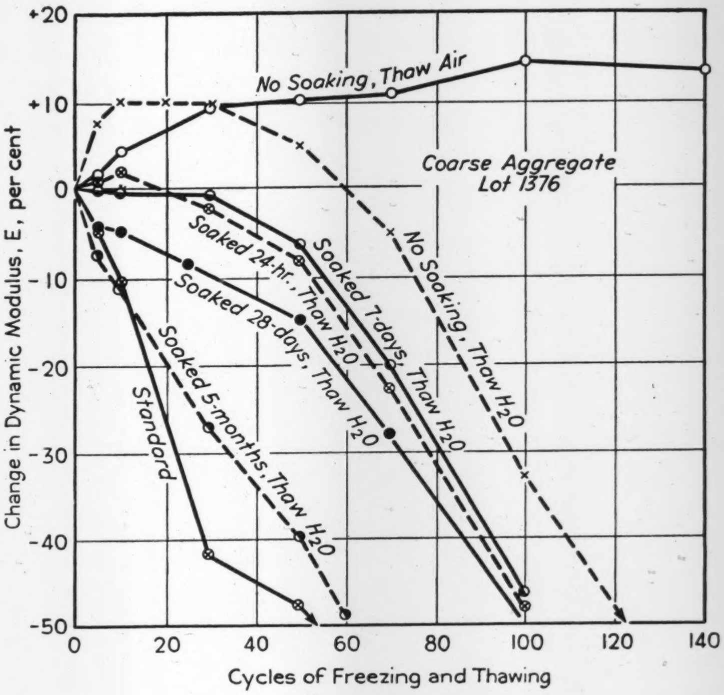

FIG. 1.—Effect of Moisture Contained in Concrete.

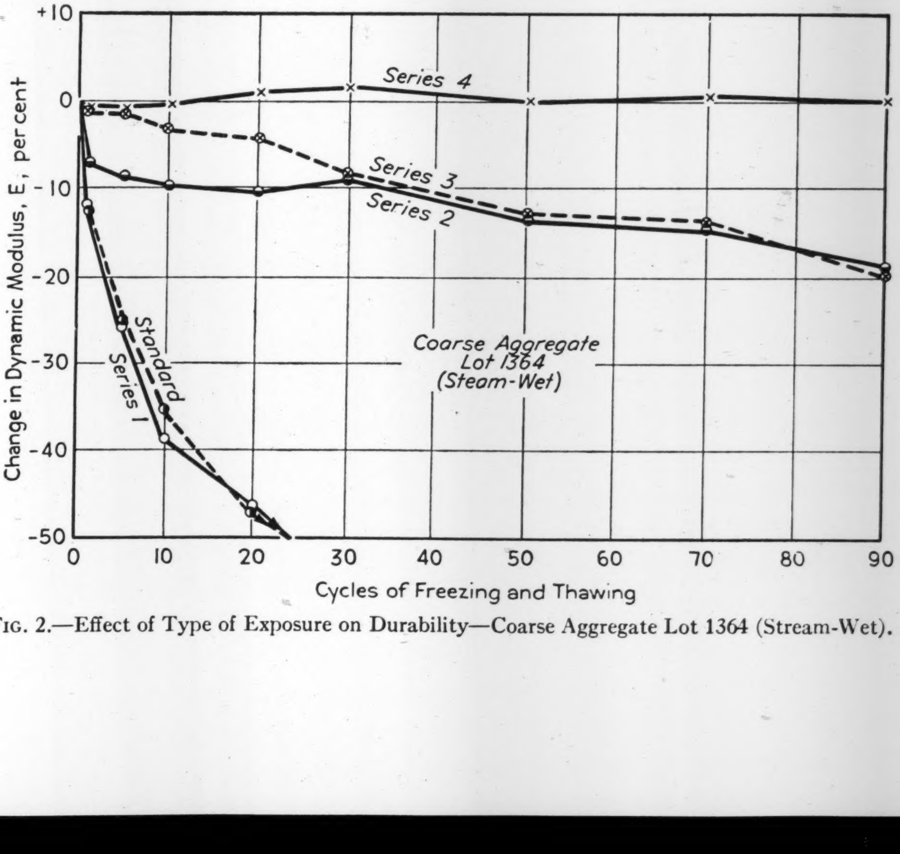

FIG. 2.—Effect of Type of Exposure on Durability—Coarse Aggregate Lot 1364 (Stream-Wet).

entirely satisfactory conditions. Generally, disintegrated sections are on terrain where opportunities for natural drainage are not good—in cuts, on side hills where ground water from above collects on and under the slab, on low, flat sections with inadequate side drains, on soil that retains water. On the other hand, the good sections of a doubtful gravel are generally on terrain where drainage is reasonably good.

All of this suggests that the moisture condition of the concrete at the time freezing and thawing takes place is of outstanding importance. It suggests that the time of year when construction is done, the curing conditions that exist after construction, and the attention paid to drainage may be the key to the solution of the problem.

The late Ira O. Baker used to say, when teaching his classes in highway engineering at the University of Illinois, that good drainage represents the first, second, and third principles of good highway construction. Modern design and construction methods, it seems to me, have, in many cases, neglected these "three" principles more than is justified. More rigid adherence to them, it seems reasonable to hope, would go further toward solving many of the problems of concrete pavement durability than some of the more spectacular panaceas.

The advantages to be gained by having concrete in a less-than-saturated condition when subjected to freezing and thawing is demonstrated by laboratory data as well as field observations. The accompanying Fig. 1 shows results of freezing and thawing of chert gravel concrete beams which, after curing in the moist room for 60 days, were dried to approximately constant weight at a temperature of about 100 F. over a period of five or six weeks. The specimens were divided into several groups and, prior to freezing and thawing, were soaked for the periods indicated. Freezing was in air at -20 F. for about 7 hr. and thawing was at 40 F., in air or water as indicated, for about 17 hr. The greater resistance of the drier concretes as compared with the more nearly saturated ones is self-evident and requires little discussion; of particular interest is the *increase* in modulus of elasticity with freezing and thawing of the concrete which had no contact with water after having been dried.

The accompanying Fig. 2 gives further data pertinent to this discussion. The concrete beams were manufactured using a stream-wet chert gravel. Specimens were divided into four groups, all of which were cured in the moist room for 28 days. After this initial curing, further treatment prior to freezing and thawing was:

Series 1—33 days in water at room temperature.
Series 2—28 days in water and 5 days in air at room temperature.
Series 3—28 days in air and 5 days in water at room temperature.
Series 4—33 days in air at room temperature.

Freezing was in air at -20 F. for about 7 hr. and thawing was at 40 F. for about 17 hr. Specimens of series 1 and 3 were thawed in water; those of series 2 and 4 were thawed in air. In all cases a 5-day "rest period" at room temperature intervened after each 5 consecutive cycles of freezing and thawing. Specimens thawed in water (series 1 and 3) were "rested" in water; specimens thawed in air (series 2 and 4) were "rested" in air. The effect of the rest period is not shown in this diagram.

Comparisons of the results of series 1 and 3 are of particular interest. While all thawing and "resting" occurred in water, the 28-day drying period for series 3 specimens, even though followed by a 5-day soaking period, vastly improved their resistance over series 1 specimens. Specimens of series 2 and 4, being thawed and rested in air, gave the expected high resistance to freezing and thawing.

It seems evident that it is the moisture condition of the chert gravel in the concrete that is of principal importance. This belief is supported by the data shown in the accompanying Fig. 3. The gravel was from the same source as that used for the tests in Fig. 2. The gravel described as "100 per cent saturated" was stream-wet. The conditions of 75, 50, and 25 per cent saturation were obtained by drying to a predetermined weight; during the drying process the gravel was continuously stirred in a gentle current of warm air. The concrete was cured 28 days in the moist room followed by 28 days in water. The freezing-and-thawing cycle was 17 hr. in air at 0 F. and 7 hr. in water at 40 F.

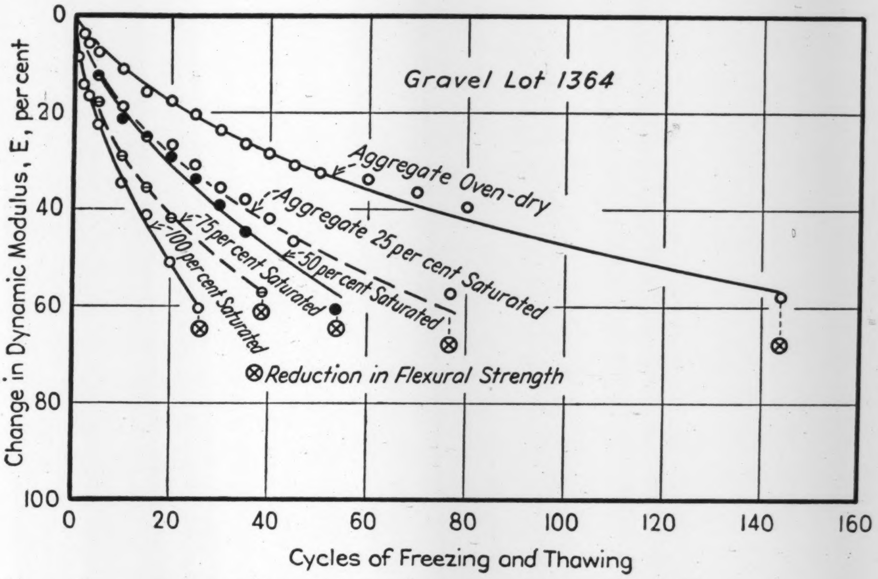

FIG. 3.—Effect of Moisture on Coarse Aggregate Lot 1364.

The data shown in Fig. 3 may be of only academic interest because of the obstacles, economic and otherwise, to furnishing a dried gravel. Further, unless precautions are taken to provide adequate drainage, the treatment may represent only a delaying action. The suggestions to be gained from the data in Figs. 1 and 2 appear to offer most promise of a practical solution.

As stated at the outset, chert gravels vary greatly in characteristics and, as has just been shown, the resistance of chert-gravel concrete to freezing and thawing varies greatly depending on conditions. These facts and observations of concrete in service suggest that different levels of prevailing winter temperatures are critical for different chert gravels and different chert-gravel concretes. An investigation just now being started by the National Sand and Gravel Association Research Foundation at the University of Maryland has for its purpose the development of information on critical freezing temperatures for concrete made with different chert gravels and cured under different conditions.

MESSRS. E. O. AXON,[4] T. F. WILLIS[4] AND F. V. REAGEL[4] (*authors' closure by letter*).—In general, it would seem that the additional evidence submitted by the gentlemen discussing this paper is corroborative of the results obtained by the authors. In the paper, it was pointed out that addition of sufficient cement to make the flexural strength of concrete, containing inferior coarse aggregate and vinsol treated cement, equivalent to that of similar plain concrete, might improve the durability sufficiently to justify the use of such a combination. Mr. Wuerpel's experience would indicate this to have been a hope rather than a possibility.

While Mr. Walker's discussion contains little pertaining to vinsol-treated cement, it discusses several other phases of the general problem of the durability of pavements containing chert aggregate (or for that matter any aggregate) and for this reason is timely. In general our observation of pavements in service and the results of other of our laboratory tests substantiate Mr. Walker's statements. It is well to remember, however, that Mr. Walker's laboratory tests, and those of all present investigators, are made using an arbitrary accelerated test procedure. Many of the conditions on which such a test is dependent for results differ considerably from the conditions provided by natural weathering. Pending direct correlation of the results of any laboratory test with behavior of similar concrete in service, categorical acceptance of the indications of such results is risky.

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[4] Associate Engineer, Chief of Research Division, and Engineer of Materials, respectively, Bureau of Materials, Missouri State Highway Dept., Jefferson City, Mo.

SIMPLIFIED TEST FOR EVALUATING THE EFFECTIVENESS OF CONCRETE MIXERS

By S. P. Wing,[1] Valens Jones,[1] and R. E. Kennedy[1]

Synopsis

A simple test for evaluating batcher and mixer performance has been developed from a study of more than 2000 mechanical analyses of fresh mortar and of a much smaller number of chemical analyses and compressive strength tests from 220 batches of concrete mixed in eight different mixers. The range in weight per cubic foot obtained by making specific gravity determinations on three samples of air-free mortar screened from fresh concrete is used as an index of mix uniformity. It was found that the initial range in weight varied from 6 to 25 lb. immediately after the mixer was charged but was reduced to 0.6 to 1.5 lb. after 15 to 25 revolutions of the mixer drum. Uniformity of mortar after a given interval of mixing is found to be approximated by a semi-emperical equation. Average ranges in unit weight are found to correlate with average ranges in water-cement ratio and strength, and a unit weight range of 1 lb. corresponds to a strength range of approximately 5 per cent for cylinders from the same batch. Applied to batch-to-batch control of concrete manufacture, test results indicate with 15 to 25 revolutions that the concrete from batch to batch is 25 to 50 per cent more variable than that indicated within the batch. Although the unit weight test does not give as much information as more complete methods of wet mix analysis, it will be found adequate and time saving for ordinary control of mixing.

"One thing needed," according to a recent article in the technical press,[2] "is a readily performable test procedure for determining whether concrete is thoroughly mixed."

The American Concrete Institute[3] published in 1939 a description of the Patch efficiency test. In Mr. Patch's hands the method proved adequate not only to determine when concrete was thoroughly mixed but also to determine needed improvements in the batching and mixing procedures. However, in discussion certain questions were raised as to the interpretation of results and limitations of his method. Since the Bureau of Reclamation at the time had three other large dams under way on which the method was being used, arrangements were made for a systematic series of experiments to clarify the issues. The results confirmed the merits of the original method but at the same time suggested a simpler test, which, while not as comprehensive, can be performed at a substantial saving in time. In addition it was found possible to express results in readily interpretable terms which are in accord with the procedures set out in the A.S.T.M. Manual on Presentation of Data.[4]

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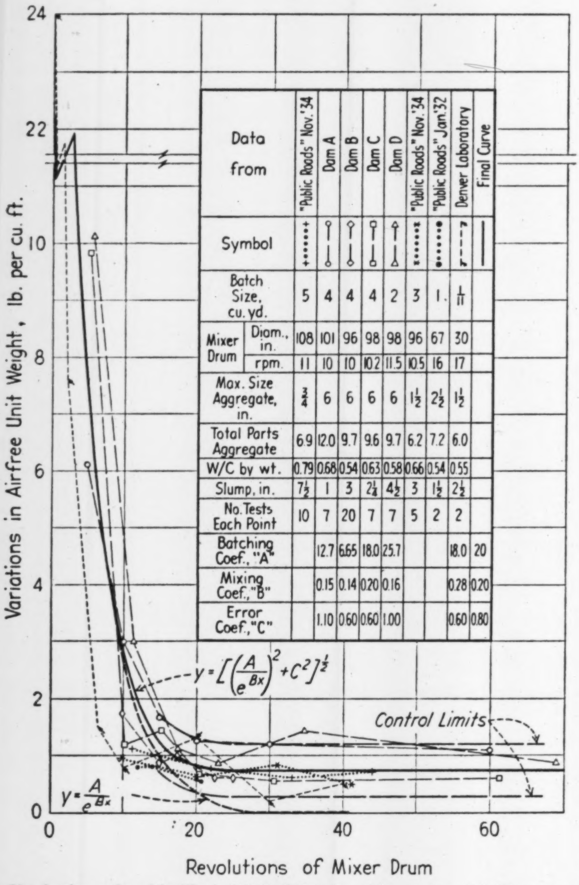
[1] Engineer, Associate Engineer, and Assistant Engineer, respectively, U. S. Bureau of Reclamation, Denver, Colo.

[2] Stanton Walker, "Quality Control of Concrete," *Pit and Quarry*, February, 1943, p. 76.

[3] O. G. Patch, "Mixer Efficiency or Mortar-Mix Tests," *Proceedings*, Am. Concrete Inst., Vol. 35, pp 173 to 180-47 (1939).

The new test cannot be expected to replace earlier methods of wet batch analyses[3,5,6] where complete information concerning the distribution of each of the materials entering into a batch is required. It is believed, however, that it will be found adequate and practical for controlling mixing and batching performance on most jobs.



Data from	"Public Roads" Nov. '34	Dam A	Dam B	Dam C	Dam D	"Public Roads" Nov. '34	"Public Roads" Jan. '32	Denver Laboratory	Final Curve
Symbol	+······+	○—○	◇—◇	□—□	△—△	×······×	●······●	- - -	—
Batch Size, cu. yd.	5	4	4	4	2	3	1.	$\frac{1}{11}$	
Mixer Drum Diam., in.	108	101	96	98	98	96	67	30	
Mixer Drum rpm.	11	10	10	10.2	11.5	10.5	16	17	
Max. Size Aggregate, in.	$\frac{3}{4}$	6	6	6	6	$1\frac{1}{2}$	$2\frac{1}{2}$	$1\frac{1}{2}$	
Total Parts Aggregate	6.9	12.0	9.7	9.6	9.7	6.2	7.2	6.0	
W/C by wt.	0.79	0.68	0.54	0.63	0.58	0.66	0.54	0.55	
Slump, in.	$7\frac{1}{2}$	1	3	$2\frac{1}{4}$	$4\frac{1}{2}$	3	$1\frac{1}{2}$	$2\frac{1}{2}$	
No. Tests Each Point	10	7	20	7	7	5	2	2	
Batching Coef., "A"		12.7	6.65	18.0	25.7			18.0	20
Mixing Coef., "B"		0.15	0.14	0.20	0.16			0.28	0.20
Error Coef., "C"		1.10	0.60	0.60	1.00			0.60	0.80

FIG. 1.—Average Variations in Air-Free Unit Weight (Averages of Ranges from Three Tests) *versus* Revolutions of Drum for Various Sizes of Mixers.

UNIT WEIGHT MIXER PERFORMANCE TEST[7]

(Specific gravity method)

Three samples of concrete, each sufficient to yield about 900 g. of mortar, are taken from different parts of a single batch. The samples may be taken while the mixer is discharging, or from the top of the batch while the mixer is stopped. In either case, a standard procedure should be followed and the sampling points should represent extreme variations in uniformity.

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[4] A.S.T.M. Manual on Presentation of Data, Am. Soc. Testing Mats. (1943). (Issued as separate publication.)

[5] W. M. Dunagan, "A Method of Determining the Constituents of Fresh Concrete," *Proceedings*, Am. Concrete Inst., Vol. XXVI, pp. 202-210 (1930). Discussion, pp. 670-698, describes several methods by different investigators.

[6] W. A. Blanchette, "Analysis of Fresh Concrete," *Concrete*, June, 1934, p. 17, and August, 1934, p. 13.

[7] "Variability of Mortar in Concrete," *Concrete Manual*, Fourth Edition, U. S. Bureau of Reclamation, pp. 431-438 (1942). The Patch mortar test was included in previous editions of the Manual.

After screening the samples through a $\frac{1}{4}$-in. sieve, they are weighed in air, and the weight of water displaced by each sample is determined either by the pycnometer method or by the immersion method such as used in the Patch test. The sample is stirred or agitated to free all entrapped air.

The air-free unit weight of each sample is then determined from the equation

$$w = \frac{a}{c + a - b} 62.36$$

where:

a = the net air weight of the mortar sample,

b = the weight of the sample and water added to fill the pycnometer, and

c = a calibration constant for the pycnometer and is equal to the net weight of the pycnometer full of water.

It is necessary to carry the solution of the equation to four significant figures. The difference between the maximum and minimum values is the range in unit weight and is the index for variability of the batch.

Precision of the work should be such that the range from sets of three samples taken from several thoroughly mixed batches should average less than 0.9 lb.

Tentatively[7] the Bureau has set a maximum range of 2.3 lb. from a single test as a standard for well-mixed concrete. If greater variability is found and is substantiated by a check test, additional mixing is required.

TEST DATA

The principal data of this paper are summarized in Fig. 1 which will be discussed later.

At three of the projects seven different batches of concrete were tested by taking a set of three samples from each batch after mixing for $\frac{1}{2}$, 1, $1\frac{1}{2}$, 2, 3, and 6 min. After the batch arrived at the forms but before it was compacted, an additional set of samples was taken. At the 1- and 3-min. sampling periods, additional samples were obtained for making compression cylinders and chemical tests. Five batches of concrete, characterized as regular, rich, lean, wet, and dry, were tested by taking six sets of three samples from the top of each batch after 1 and 3 min. of mixing.

A fourth dam supplied a series of 115 batches tested after either 1, $1\frac{1}{2}$, 2, $2\frac{1}{4}$, or $2\frac{1}{2}$ min. of mixing. Three samples were obtained, after the given period of mixing, from the first, middle, and last of each batch as the mixer was discharging.

Tests published in *Public Roads*[8] in 1932, on a 27E paver, consisted of five 25-lb. samples from each batch collected by pans placed on the subgrade below the skip. Six batches, in sets of two, were tested for mixing periods of 45, 55, and 75 sec. after all solid material had entered the batch.

Wet analyses were made for all ingredients in the above tests.

The 1934 tests[9] with 84S and 126S mixers were made on samples obtained while the mixer was discharging. Four mixing times from 55 sec. to 3 min. and 55 sec. were used for 20 batches in the 84S mixer and for 40 batches in the 126S mixer. Analysis of unit weight variation was computed from the published data.

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[8] T. C. Thee, "Effect of Size of Batch and Length of Mixing Period on Rate Production and Quality of Concrete Mixed in Standard 27E Pavers," *Public Roads*, Vol. 12, No. 11, January, 1932, pp. 269–288.

[9] William A. Blanchette, "Effect of Mixing Time on Quality of Concrete Produced in Large Mixers," *Public Roads*, Vol. 15, No. 9, November, 1934, pp. 217–230.

Finally, the performance curve for a $3\frac{1}{2}$S mixer was obtained by sampling two batches at eight intervals up to 40 revolutions, testing by the pycnometer method.

Data on water-cement ratio, sand-cement ratio, and strength are given later with discussions of results.

DISCUSSION

The function of a concrete mixer is to mingle within the briefest time the component materials entering a mix into a homogeneous mass to obtain reasonably uniform properties in the hardened concrete. Practically this means that the water-cement ratio of different parts of the batch, or some index closely related to it, must approach uniformity. Air-free unit weight of mortar is such an index since its variation largely reflects changes in water content.

Analysis of wet ball mills of different diameters has shown that their performance tends to be similar when operated at the same percentage of the "critical speed."[10] Concrete mixers are ordinarily operated at about equal percentages of critical speed, so it seems reasonable to consider mix variability as a function of mixer revolutions so that a direct comparison of the performance of the different size mixers is possible. For mixers of equally efficient design, the time required to produce a given degree of uniformity will vary as the square root of the diameters, a 100-in. mixer requiring 80 per cent longer mixing time than a 30-in. mixer.

Equation for Mix Variability:

Concrete materials entering a mixer from the loading skip or hopper have weights per cubic foot of solid volume of about 60, 160, and 200 lb. for the water, sand, and cement. If weights of three samples be determined immediately after a mixer is charged, a difference in weight of from zero to 140 lb. is possible, depending on the material obtained in the samples. Actually such extreme ranges are not probable since the batching process in itself effects a certain amount of premixing. Each revolution of the drum decreases the range in unit weight by moving a certain amount of material across and from end to end of the mixer. In measuring variation in unit weight two independent causes of variations are present, true variations attributable to the batching procedure and mixing process and apparent variations due to experimental error. The two sources of variation can be combined to form the general equation

$$Y = \left[\left(\frac{A}{e^{Bx}}\right)^2 + C^2\right]^{\frac{1}{2}}$$

where Y is the range of air-free unit weight for three samples, x is revolutions of the drum, and A, B, and C are batching, mixing, and error coefficients, respectively, and e is 2.718, the base of natural logarithms.

Referring to Fig. 2, a test on a 4-yd. mixer, immediately after the solid materials are in the drum (one to two revolutions) the average range in weight, determined from seven different batches, gave an average batching coefficient of about 13 lb. Any radical change in batching procedure or method of taking samples would be reflected in this coefficient.

The second term of the general equation represents experimental error and if this be neglected the equation for true variability can be written $y = \frac{A}{e^{Bx}}$, or $\log_e y = \log_e A - Bx$, from which $\frac{dy}{dx} = -By$. The equation shows that the var-

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[10] W. H. Coghill and Fred D. Devaney, "Ball Mill Grinding," *Technical Paper No. 581,* U.S. Bureau of Mines (1937).

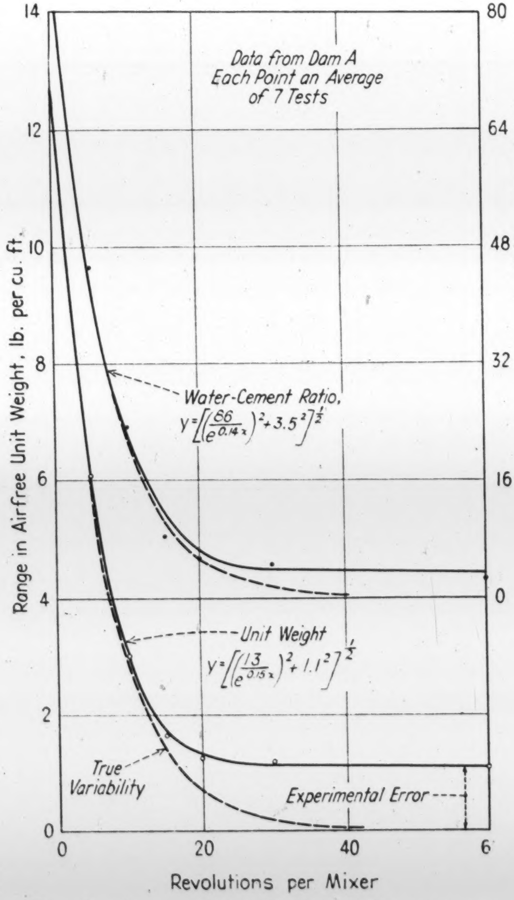


FIG. 2.—Average Range in Unit Weight Forms a Smooth Curve.

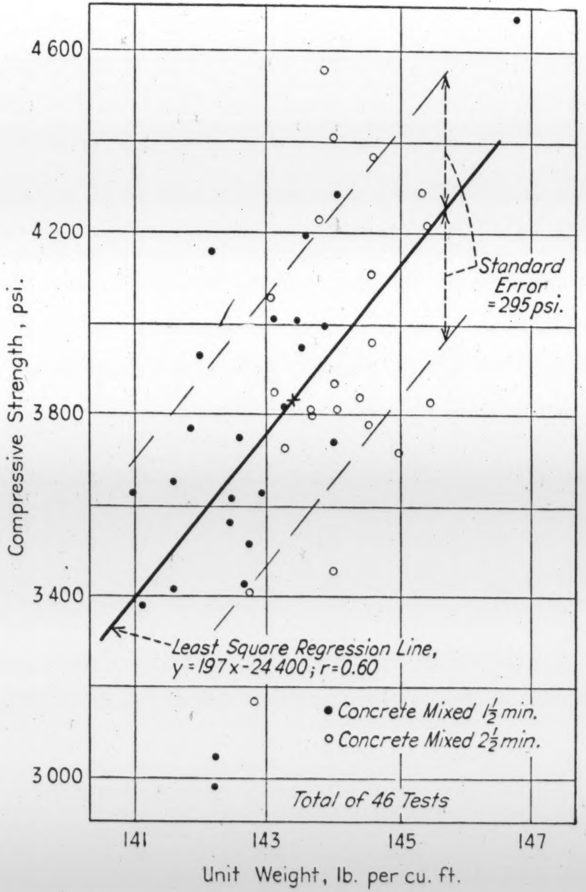


FIG. 3.—Correlation Between Compressive Strength and Air-Free Unit Weight Is Highly Significant.

iability (y) for x revolutions is proportional to the initial variability found just after batching, and that the rate of reduction per revolution is a constant fraction of the variability existing at the moment. For dam A, Fig. 2, the rate of reduction is 0.15 or 15 per cent of the unit weight range at the point considered.

At about 15 revolutions, in Fig. 2, the curve of range in unit weight *versus* mixer revolutions rapidly flattens and becomes horizontal, experimental error obscuring any further reduction in unit weight range. At 20 revolutions, where true variability shown by the dashed curve is about 60 per cent of the experimental error of 1.1 lb., the measured variability (heavy curve) is only 16 per cent more than the experimental error. Personal equation and inherent errors of the test method are included in the constant C.

Compressive Strength Related to Air-Free Unit Weight:

From the shape of the computed curves in Fig. 2 it is evident that range in unit weight is correlated with range in water-cement ratio and consequently with strength. The correlation of strength with average unit weight is shown in Fig. 3. There were 46 batches tested, some after $1\frac{1}{2}$ min. mixing and some after $2\frac{1}{2}$ min. A statistically significant correlation of 0.60 was found. The least square line fitted to the graph shows 197 psi. or 5 per cent increase in strength for each one pound increase in unit weight. Since other data from the same job indicate that 1 per cent change in water ratio changes strength about 60 psi., a change of 1.0 lb. in unit weight is equivalent to a variation of about 3.3 per cent in the water-cement ratio.

Control Chart Analysis of Mixer Performance:

Figure 4 shows control charts for batches tested on 22 consecutive shifts in a one week's period, the tests being made on a 4-yd. mixer after 25 revolutions. Samples were caught from the first, middle, and last of the discharge and completely analyzed by the Patch method, so that variability of unit weight, water-cement ratio, and sand-cement ratio are comparable. The two graphs at the top constitute control charts for average unit weight (control from batch to batch) and range of unit weight (control within the batch). The control limit for range is equal to the average range for the batches considered times a factor obtained from Table I of Supplement B, of A.S.T.M. Manual on Presentation of Data. For a range of three tests, the control limit is in this case 0.63 × 2.57 or 1.62 lb. Briefly, values exceeding the control limit are abnormal and cause for their variability can probably be found if search is made. For values within the control limit, experience has generally shown that search for individual causes of variation will be fruitless. The control limits for average unit weight, having three tests to the batch, are equal to 1.02 times the average range within the batch, the factor again being obtained from the A.S.T.M. Manual.

The chart for average unit weight indicates definite lack of control. Investigation of data for the month from the job indicated that weighing of the separate ingredients was well controlled but that moisture content of the sand fluctuated enough that a maximum change of 5 lb. in unit weight could have been obtained. Evidently part but not all of this variation was controlled at the mixer by changing the added water to obtain uniform consistency.

Only one point being outside the chart for range in unit weight indicates good control within the batch. Moreover, the fluctuations largely reflect experimental

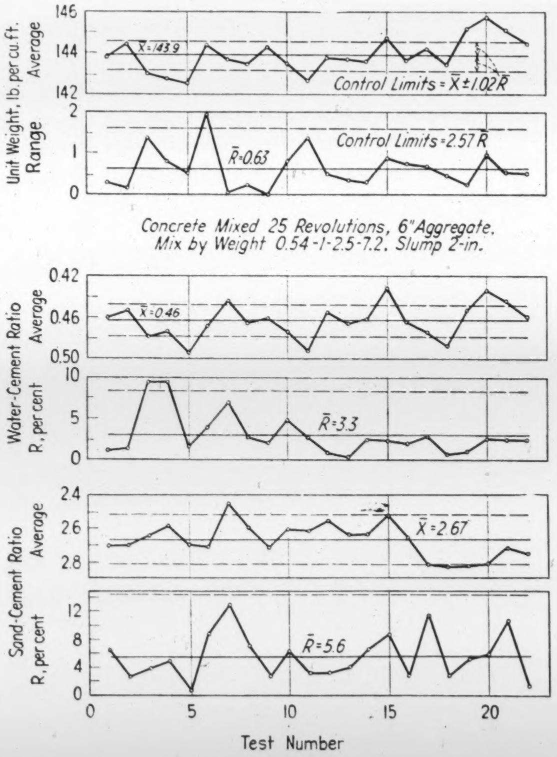


FIG. 4.—Control Charts Show Lack of Uniformity from Batch to Batch.

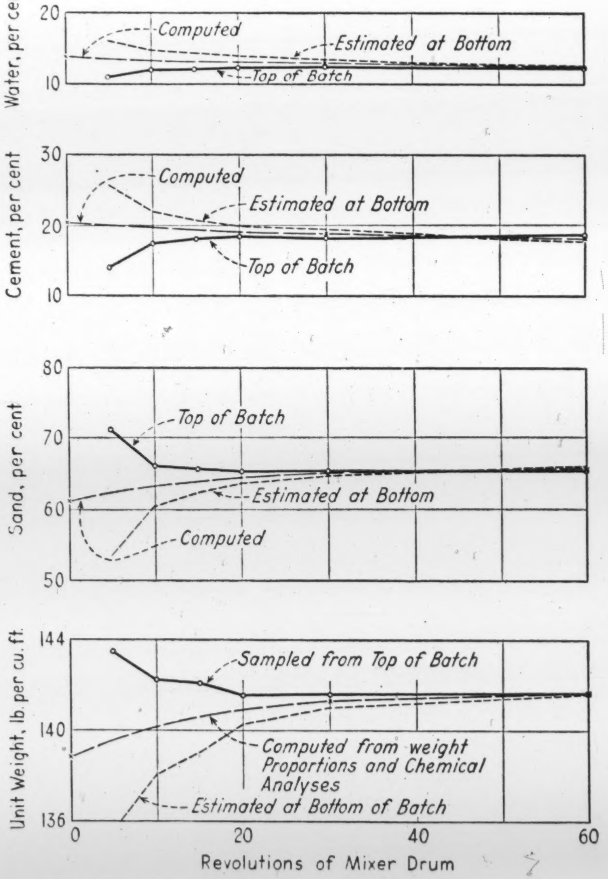
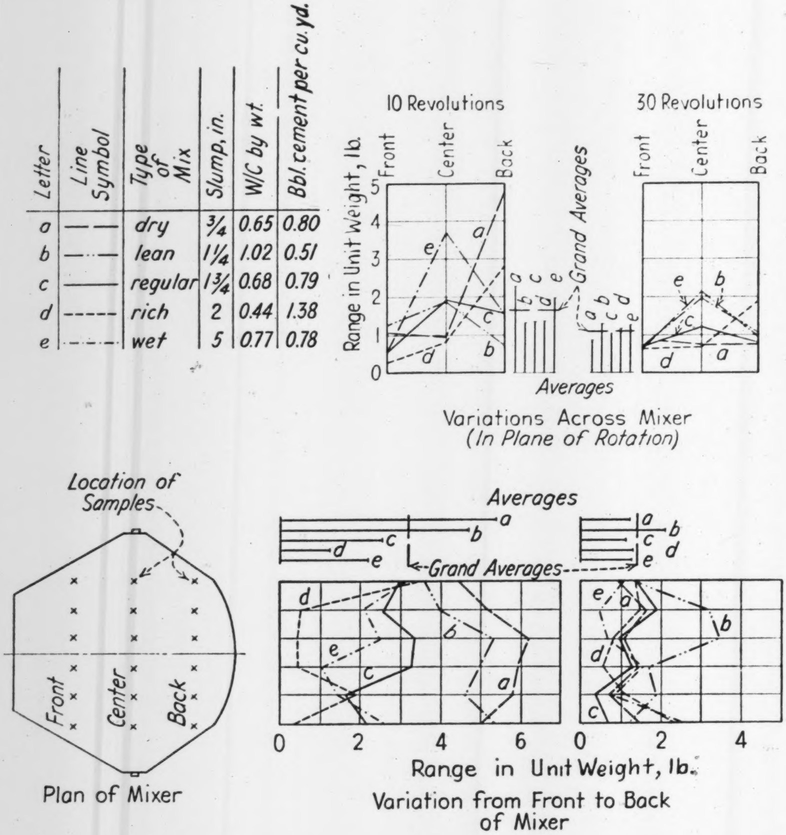


FIG. 5.—Properties of Air-Free Mortar from Dam A.

error which on this job averaged about 0.6 lb.

Control limits for water-cement ratio and sand-cement ratio, with the same probability as those used for unit weight, were also computed from average variations within the batch. The sand-cement ratio, having a larger percentage variation within the batch, likewise requires wider limits for the averages from batch to batch. The relationship of these various indices of uniformity control will be described later.



Letter	Line Symbol	Type of Mix	Slump, in.	W/C by wt.	Bbl. cement per cu. yd.
a	— —	dry	3/4	0.65	0.80
b	—··—	lean	1 1/4	1.02	0.51
c	———	regular	1 3/4	0.68	0.79
d	-----	rich	2	0.44	1.38
e	-·-·-	wet	5	0.77	0.78

FIG. 6.—Variation in Range of Unit Weight for Widely Differing Concrete Mixes, Dam A.

Variations Within the Batch:

Figure 5, derived using the Patch method for determining water-cement and sand-cement ratios and from chemical tests of samples taken from the top of the batch, each point an average of three tests on seven batches, shows in a fundamental way what is actually taking place in the batch. The bottom graph shows that the mortar at the surface of the batch gradually decreases from a unit weight of 144 lb. at 5 revolutions to 142 lb. at 60 revolutions. On the other hand, unit mortar weight in the bottom of the mixer and the average of the whole batch is estimated to increase to a value above that computed from the initial quantities. Two factors are probably responsible. First, grinding of coarse aggregate produces more sand which increases the quantity of high specific gravity material in the mortar. Second, in separating mortar from concrete by wet screening, some paste (water and cement) is left on the coarse aggregate whereas practically all the sand is removed. The initial average weight shown on the graph is that computed from specific gravities of the materials. The curves showing weights of the material in the bottom of the mixer are computed to agree with the average. The percentages of water, cement, and sand reflect the change in unit weight and have other causes of variation which are discussed later. Qualitatively the graphs agree with the tests by Waugh,[11] who found after 30 revolutions, in four out of six tests, a small increase in total sand.

Figure 6, mixer A, shows the results of determining the unit weight at 18 points located in the top of the batch for mixing 10 and 30 revolutions and for five different batches of widely different characteristics. Ranges of unit weight for sets of three are plotted on the diagrams and will repay study. The two bottom graphs show six ranges taken from the six different positions in the mixer, as shown in the figure, the averages for the five types of mixes being plotted above. In the two graphs at the top, the range of six samples is converted to an equivalent range for a set of three. These ranges are indicative of the variation across the mixer at the front, center, and back. Considering the group of mixes as a whole from front to back the average range reduced from 3.2 lb. at 10 revolutions to 1.4 lb. at 30 revolutions. On the other hand, across the mixer even with as few as 10 revolutions, the average range is reduced to 1.6 lb., becoming 1.0 lb. at 30 revolutions. Evidently the mixer is more efficient at mixing materials in a plane parallel to rotation than it is in moving material from end to end. If it be assumed that initial variability was the same in both directions, then, since the mixing coefficient B is proportional to the logarithms of the ratio of the initial to final variability for any given amount of mixing, by computation, the mixing coefficient in the plane of rotation is 0.22 as compared to 0.15 at right angles to it. At 10 revolutions the mortar is more variable at the back of the batch but at 30 revolutions mortar in the center is more variable, suggesting a design with a narrower drum or a change in blading as a possible improvement for this particular case.

Precision and Accuracy:

Precision of the average of several observations is based on some measure of variability that describes the manner in which the individual values cluster about the average value. Mean variation, range, standard deviation, and coefficient of variation are used for this purpose and are more adequately described elsewhere.[4] However, each of these indices of variation under controlled conditions has a precision, measured in per cent, which is approximately inversely proportional to the square root of the number of observations used to obtain the average index. The relation of precision to the number of observations is approximated from the standard deviation times $1/\sqrt{2(n-1)}$ where n is the number of tests.[12] In other words, the standard deviation of three tests in a group has a standard error of about 50 per cent of the value of the standard deviation. If several ranges or standard deviations are averaged, the standard error is reduced in proportion to the square root of the number of sets so that the standard error of an average of four ranges of sets of three is about 25 per cent, seven ranges, 20 per cent, and 25 ranges, 10 per cent. This means that the average range of unit weight of 0.6 lb., shown in Fig. 5, would be found within the range of 0.6 ± 0.06, two thirds of the time if sets of 25 were repeated under controlled conditions. A range of 0.6 lb. for three determinations of unit weight corresponds to duplicate determinations of specific gravity with a spread of 0.006, a value less than the 0.01 given in A.S.T.M. Standard Methods of Sampling and Physical Testing of Portland Cement (C 77 – 40)[13] for specific gravity determinations on cement.

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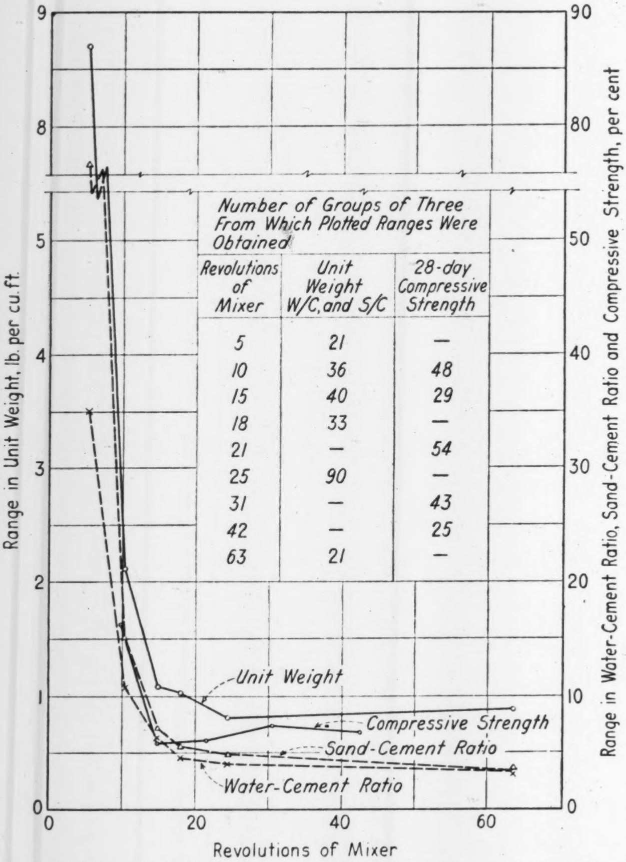
[11] W. R. Waugh, "Effect of Grinding in the Large Mixers on Aggregate Grading at Hiwassee Dam," *Journal*, Am. Concrete Inst., Vol. 14, No. 1, September, 1942, pp. 9–20.

[12] Mordecia Ezekiel, "Methods of Correlation Analysis," John Wiley and Sons, Inc., New York, N. Y., p. 26 (1930).



Number of Groups of Three From Which Plotted Ranges Were Obtained

Revolutions of Mixer	Unit Weight W/C, and S/C	28-day Compressive Strength
5	21	—
10	36	48
15	40	29
18	33	—
21	—	54
25	90	—
31	—	43
42	—	25
63	21	—

FIG. 7.—Curves of Four Measures of Variability Have Similar Shapes.

Accuracy of a test expresses how close the measured value approaches the true value. Accuracy of unit weight determined by specific gravity methods is generally high. High accuracy is not the only criterion of the usefulness of a test. Of more importance is the relationship of the test to the service characteristics for which the test is desired, in this case strength, strength being the principal criterion for job control. Numerous series of tests show that if the range in unit weight within a batch is kept within an average of 0.6 to 0.8 lb., the corresponding ranges in strength will be about 5 per cent or have a coefficient of variation of about 3 per cent.

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[13] 1942 Book of A.S.T.M. Standards, Part II, p. 33.

Required Mixing Period:

It is not the purpose of this paper to specify the proper amount of mixing for any given job, but certain guiding principles may well be pointed out. Economy demands that the uniformity required within a single batch of concrete should vary with the uniformity found possible for the job as a whole. Tests have shown that under present practice the variation to be expected in 28-day strength of cylinders cast in the course of a job ranges between 10 and 25 per cent as expressed by the coefficient of variation. If mixing is continued to the point where only 10 per cent of this total is due to the mixing process, such concrete might be called adequately mixed, variations in workability and distribution of coarse aggregate not being considered.

When several sources of error are being considered, their squares may be added under the radical to obtain an estimate of a combined value. Thus if the coefficient of variation of strength from batch to batch is 9.0 per cent and the coefficient of variation from a single batch is 4.5 per cent; the combined coefficient of variation for the job would be $(9.0^2 + 4.5^2)^{\frac{1}{2}} = 10.0$ per cent. The variation within the batch is then equivalent to a range of strength for three cylinders of $1.69 \times 4.5 = 7.5$ per cent. In Fig. 7 an average range of 1.0 lb. in unit weight corresponds to an average range in water-cement ratio of 5 per cent at 20 revolutions of the mixer, and for this much mixing, a range of strength for three cylinders from the same batch of 6.0 per cent was obtained on the average. On one job, batches tested at the mixing plant were also tested after being dumped from the bucket into the forms but before consolidation. The average range in unit weight for five batches was 1.1 lb. at the plant after 6 min. mixing and 1.25 lb. at the forms. On two other jobs a regular batch mixed about 25 revolutions was sampled at the form just before sampling a similar batch at the mixing plant. The average of 15 mortar tests from the forms was 1.1 lb. range in unit weight, whereas the average of similar batches at the plant was 1.0 lb. The differences are not conclusive in either case but show a trend toward decrease in uniformity of the concrete with handling.

Other properties of a mix, particularly workability and maximum size of aggregate can be expected to change the mixing coefficient. Blades suitable for $\frac{3}{4}$-in. aggregate and a 7-in. slump cannot be expected to perform efficiently in a dry, lean mix where their work resembles plowing. None of the tests was adequate to determine the effect of slump, but combined they indicated that a change of 1-in. in slump of moderately dry concrete would change the mixing coefficient by 15 per cent. This would change the required mixing period by about the same amount.

TESTS BY OTHERS

Method of Averages:

Most early tests were based on the assumption that average strength of cylinders from a batch would increase with increased mixing and that when this increase became negligible the concrete was adequately mixed. On this basis Besson[14] in 1933 made a critical review of earlier mixer tests and presented his own results comparing 1- and 2-min. mixing time using two $\frac{3}{4}$-yd. of mixers running at 12 rpm. He concluded for his own mixers, "It is a waste of money to mix concrete 2 min.," and stated "a failure to find in the review of published data any test supporting a 2-min. mix." However, when some of the tests which he reviewed were analyzed by the unit-weight method, they indicated a definite decrease in variability with increased mixing. Such tests and others similar to it do not indicate whether all constituents of the concrete are uniform to an acceptable degree from one part of the batch to another.

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[14] F. S. Besson, "Over a Minute Adds No Strength," *Engineering News-Record*, February 9, 1933, pp. 183-185.

If sufficient samples are taken from a batch to be representative of the batch as a whole after 10 to 20 revolutions of mixing, the *average* 28-day strength will be much the same since a high water-cement ratio at one point tends to be compensated by a low value somewhere else. However, with longer mixing periods, workability and the average 28-day strength are almost certain to increase slightly because of grinding increasing the specific surface of the cement.

Wet Analysis:

Harrison in 1928,[15] Thee in 1932,[8] and Blanchette in 1934,[9] all of the Bureau of Public Roads (now the Public Roads Administration), made tests at different times on 27E to 126S mixers, at intervals of from about 45 sec. to 4 min. (40 revolutions). Complete wet mix analyses were made with corresponding strength tests. While space will not permit giving a detailed analysis of their results, in general they concluded that increasing the mixing time beyond 1 min., or about 10 to 15 revolutions, did not increase the uniformity of results or increase the strength. However, when analyzed on a unit weight basis their tests showed a general trend toward both lower ranges in unit weight and strength with increasing revolutions. For example, with an 84S mixer the unit weight range at 10 revolutions was 0.9 lb. and strength range 9.0 per cent, whereas at 41 revolutions the values were 0.5 lb. and 6 per cent. The corresponding coefficient of variation of strength would be about $5\frac{1}{2}$ and $3\frac{1}{2}$ per cent, respectively.

The Patch Test:

The unit weight test for mixer performance is the result of a study of the original Patch test of mixer efficiency[3] in which ranges of water-cement and sand-cement ratios were used as indices of variability. Since the Patch method requires an additional weight measurement after wet-screening the mortar over a No. 100 sieve, it is reasonable to expect that water-cement and sand-cement ratios together will show objectionable fluctuation in more cases than unit weight alone. Being composed of three constituents of widely different specific gravities, there are obviously many combinations that will produce a mortar of the same unit weight.

By observing the general shape of the curves for variability of unit weight, water-cement and sand-cement ratios, and compressive strength in Fig. 7, it would be reasonable to expect that any of the four factors used singly would give comparable results with the use of proper limits. A study of variation in strength with variation in unit weight gave a correlation ratio of 0.60. A similar correlation with water-cement ratio gave a ratio of 0.49. However, the difference in favor of the unit weight is without statistical significance. The close parallelism of curves for range of unit weight and water-cement ratio in Fig. 2 is indicative of the close relationship of these two factors. A similar relationship is shown in Fig. 4 between averages for unit weight and water ratio, the correlation ratio being 0.79 by least squares. Eight batches out of 22 are shown to exceed the control limits for average unit weight and water-cement ratio, six being common to both.

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[15] J. L. Harrison, "The Effect of the Length of the Mixing Period on the Quality of the Concrete Mixed in Standard Pavers," *Public Roads*, July, 1928, pp. 93–111.

The Patch test gives data from which the amounts of sand, cement, and water in the batch may be computed—data which are often required in a complete analysis of mixer performance. While the absolute accuracy of such determinations is low on account of assumptions of specific gravities of materials and amount of fine sand in the mortar which may change both within the batch and from batch to batch, the relative values are generally all that are required.

CONCLUSIONS

1. The unit weight test is a simple test which is well adapted for determination of the amount of mixing necessary to produce a desired degree of uniformity. It provides coefficients indicative of the batching and mixing efficiencies and indicates where improvements can be made in the mixing procedures.

2. The unit weight method was found to be about 80 per cent as efficient as the Patch method in detecting nonuniform batches using comparable control limits of 15 per cent for water-cement ratio, 21 per cent for sand-cement ratio, and 2.0 lb. for unit weight from several sets of tests analyzed.

3. For a given uniformity and operating at the same percentage of critical speed, concrete mixers of different sizes require mixing periods directly proportional to the square roots of their diameters.

4. Air-free unit weight was found to correlate with compressive strength, a change of 1 lb. in unit weight indicating a 200-psi. change in average strength.

5. Slump has an influence on the period of mixing necessary to obtain uniformity, dryer concrete requiring longer mixing.

6. Variation of concrete properties from batch to batch was appreciably greater than that within the batch after a usual period of mixing.

7. The most economic mixing period should be determined from a consideration of variability which exists in the job as a whole.

8. The inherent nature of variability measurements requires many tests before there is certainty that small differences are real.

Acknowledgments:

This paper could not have been written without the active cooperation of the engineers of the Bureau of Reclamation, both in the field and in the Denver office. L. H. Tuthill was responsible for suggesting unit weight as the measure of mortar uniformity. Engineering research and design are under the supervision of Chief Designing Engineer J. L. Savage. All engineering is under the direction of Chief Engineer S. O. Harper. Activities of the Bureau are under the administrative supervision of Commissioner John C. Page.

DISCUSSION

Mr. Duff A. Abrams[1] (*by letter*).—This paper is a continuation of the rebuttal to my criticisms of a paper by O. G. Patch[2] on the same subject in which I showed that concrete at Grand Coulee Dam was habitually overmixed, sometimes to such an extent as to produce a sand-cement, as a result of a mistaken interpretation of data obtained in routine tests of mixers.

Overmixing of concrete is not merely an academic question. In 8-ton batches, using sand and gravel graded up to 6 or 9 in. in diameter, it is easy to overmix concrete. Overmixing not only wastes time and money but is a serious detriment, due to the large quantity of sand pulverized by the abrasion and impact of cobbles weighing up to 15 or 50 lb.

My conclusions with reference to Grand Coulee concrete were based on 2 tests. I repeatedly requested additional data; this was refused. Instead of furnishing a few dozen values from probably 25,000 routine tests then on hand at Grand Coulee, where the Patch test had been made 24 times daily for 4 yr., "arrangements were made for a systematic series of experiments to clarify the issues," and we are now furnished a few "coefficients" from "three other large dams." It will be seen that most of these tests were not made at Grand Coulee.

Summary

Few of the "issues" raised by my earlier criticisms of concrete mixing at Grand Coulee Dam were considered and none was clarified; instead the authors divert our attention to a "simplified test for evaluating the effectiveness of concrete mixers." But this test[3] is only incidental; the bulk of the paper concerns a body of doctrine on mixing and other properties of concrete to which this discussion is directed.

The paper is an obvious attempt: (1) To justify the mixing times that the U. S. Bureau of Reclamation arbitrarily wrote into specifications for large concrete dams (Boulder, Grand Coulee, Parker, Seminoe, Marshall Ford, Shasta, Friant) built in the past ten years; (2) to refute my criticism that the concrete at Grand Coulee was overmixed; (3) to give credence to the Patch test, discarded in 1941; (4) to introduce certain "improvements" in the Patch test; (5) to present a nonchalant attitude toward a change in sampling methods at Grand Coulee in 1936; (6) to find a test that does not permit one to compute the sand pulverized in the mixer; (7) to divert attention from routine mixer tests at Grand Coulee; (8) to draw a veil over many "issues" raised by my criticisms.

Insignificant and erroneous correlations were emphasized; many important statements were couched in cryptic or contradictory language.

The new test involves fallacies that do not differ much from that of Mr. Patch; the authors assumed:

That unit weight of mortar is "closely related to water-cement ratio of concrete."

Only air-free mortar need be considered.

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[1] Consulting Engineer, New York, N. Y.

[2] O. G. Patch, "Mixer Efficiency or Mortar-Mix Tests," *Proceedings*, Am. Concrete Inst., Vol. 35, pp. 173 to 180-47 (1939).

[3] The "simplified test" is so incompletely described that it is impossible to carry it out without going to the U. S. Bureau of Reclamation "Concrete Manual," 1942, which devoted seven pages to it.

That the actual unit weight of mortar is unimportant.

A zero range in unit weight of three samples, represents perfectly mixed concrete.

It is shown in this discussion that:

There is no foundation for any conclusions on relation between unit weight of mortar and water-cement ratio of concrete. Unit weight generally decreases as water-cement ratio increases, however it may increase or it may do neither.

A high-range mix may indicate imperfect mixing, but such a test is ignored unless "substantiated by a check test."

Low-range batches are always accepted, but they may be as poorly mixed as those with a high range.

The assumption of air-free mortar excludes one half of possible mixtures of water, cement, sand.

Unless the air-free mortar has the correct unit weight for the materials and mix used, and at the same time the known value for water-cement or sand-cement ratio, a zero range does not represent perfect mixing.

There are 35,000,000,000 different mixes of zero range in unit weight of three samples, only one of which is perfectly mixed.

The fallacies of the paper make nonsense of the multistoried exponential equations and statistical theories that are offered as substitutes for withheld test data, and nullify all conclusions based on the unit weight of mortar.

There was the same lack of data on materials, methods, and tests to which I have repeatedly called attention. Only one-half column was devoted to "Test Data," but about eight columns were given to exponential equations and statistical theories. All mixer data were buried in calculated "coefficients." Hypothetical curves override plotted coefficients. In Figs. 1 and 2 large experimental errors were indicated for 20, 30, 60 revolutions of the mixer drum, but no errors for 5 or 10 revolutions, although the equations of the hypothetical curves show constant errors.

Erroneous assertions and absurd conclusions arose from a failure to note the dominant influence of sampling methods on the unit weight of mortar. Regardless of when the mixer was stopped (Fig. 5) the heaviest mortar was *at the top of the batch.* It is apparent that something besides mixing was involved when we find (Fig. 6) that at 10 revolutions the same mixer was 10 times more efficient with a rich mix than with a lean one, and that longer mixing increased unit weight at Grand Coulee Dam, but decreased it at Friant Dam (Figs. 3 and 5).

Conclusions were stated that have no foundation on the tests; for example, conclusion 3 on mixing periods for mixers of different diameters. In most instances absence of important data, acknowledged lack of control, "unusual" test conditions, conflicting "data," contradictory statements concerning the tests as well as the cryptic language used make the reported values worthless as a basis for usable conclusions.

Conclusions are stated as of general application, but many (perhaps most) of the tests were made with a Bessemer-converter type of concrete mixer not used elsewhere.

Most of the tests to "clarify the issues," with reference to concrete in Grand Coulee Dam, *were not made at Grand Coulee*, but at "three other large dams." The tests given the most prominence were made at Friant (dam A) using a mix in which 21 per cent of the normal quantity of cement was replaced by pumicite, finer than cement. They were not made with the 3-in. slump used at Grand Coulee, but with a 1-in. slump. These factors lengthen the time required for a given degree of mixing uniformity. Pumicite at Friant Dam was carefully concealed from the reader.

Tests in Fig. 4 embody three important departures from Grand Coulee practice; they were not even presented as typical, but accompanied an elaborate apology that shows them to be "unusual." The tests place the performance of the mixers at Grand Coulee Dam in the worst possible light. The illogical methods used by U.S.B.R. in computing mixer efficiency have always exaggerated the small variations shown by the limited data they have released.

The new test is unscientific; the fallacies and contradictions involved leave nothing to recommend it, but if we accept it at face value it furnishes four proofs that concrete at Grand Coulee was well mixed at 1 min. This confirms my conclusions from a study of the Patch test in 1939.

My study of mixer grinding at Grand Coulee Dam shows that after mixing 15 min. there was fine sand and rock dust equivalent to 95 per cent of the weight of the cement. This confirms my former conclusion that a sand-cement was used in the foundation of the dam.

The U.S.B.R. mixer test is the result of 10 yr. of research. It is regrettable that their scientists did not give the method the little study necessary to reveal its fallacies before it was specified on the world's largest concrete dams, and entered in a "Concrete Manual" that is now offered for sale to the public.

The U.S.B.R. errors in mixer tests are much more fundamental than may appear at first sight; they go back to the sand and gravel pits, to proportioning of mixes, and affect many phases of the design and construction of a large dam. Unfortunately many of the errors came to light *after* their vast program of concrete dam building was finished.

No complete evaluation of mixing blunders at Grand Coulee can be made until the U.S.B.R. releases data from tests made during 1935 to 1939. Since they again bring their mixer test to the fore, after dropping the discussion abruptly 4 yr. ago, concrete engineers are now justified in demanding that the data be released.

History of the U. S. Bureau of Reclamation Mixer Tests

During the 10 yr. that the U.S.B.R. test for concrete mixer efficiency has been in use, it has had a somewhat checkered career:

1933–35, Method originated by O. G. Patch during concreting of Boulder Dam.

1936, Oct. 15. Patch test at Grand Coulee Dam described; states concrete was mixed up to 15 min.; *Engineering News-Record*.

1937, November. Mixing requirements based on Patch test in second Specifications (757) for Grand Coulee Dam.

1938, May. Method in Specifications (780) for Shasta Dam.

1939, January. Paper by O. G. Patch based on daily use of method on Grand Coulee Dam during preceding 3 yr.; *Journal*, Am. Concrete Inst.

1939, March. Mr. Patch gave his "mortar-mix test" as No. 1 in a list of "new methods and equipment" that came "out of the experiences gained on the great concrete building projects such as Boulder Dam and Grand Coulee Dam. . ."; *Concrete*.

1939, June. Discussion of paper by the writer, O. G. Patch, and Arthur Ruettgers; *Journal*, Am. Concrete Inst., pp. 180-1 to 180-24.

1939, July. Method in specifications (863) for Friant Dam.

1939, September. Further discussion by the writer, closure by Patch; *Journal*, Am. Concrete Inst., pp. 180-25 to 180-47.

The second paragraph of the paper begins:

"The American Concrete Institute[3] published in 1939 a description of the Patch efficiency test. . . . However, in discussion certain questions were raised as to the interpretation of results and limitations of his method."

Footnote 3 refers to *Proceedings* of the American Concrete Institute (1939), which contained my discussions. Since I was the only one to discuss the Patch paper, it is obvious that the present paper is a much-delayed and somewhat oblique reply to my criticisms. Oblique in that:

(*a*) A discussion in the A.C.I. *Proceedings* that was abruptly dropped 4 yr. ago is resumed before the A.S.T.M.;

(*b*) My criticisms were not mentioned;

(*c*) Overmixing of concrete at Grand Coulee was not mentioned;

(*d*) Mixing times specified or used at Grand Coulee were not mentioned;

(*e*) Grand Coulee Dam is not mentioned;

(*f*) My most important criticisms and all direct questions were ignored;

(*g*) My criticisms were based solely on Grand Coulee practice, whereas in the paper is notable for the inadequacy of Grand Coulee tests;

(*h*) Instead of discussing the test criticized by me, a "simplified test" is substituted;

(*i*) Nearly every paragraph of the paper seems to be a response to one of my criticisms.

I shall call attention to a few of the criticisms that were ignored and some of the responses.

After making a study of the additional tables and figures presented by Patch and Reuttgers, Senior Engineer, U. S. Bureau of Reclamation, the following were among the 13 conclusions stated in my second discussion (*Journal*, Am. Concrete Inst., Supplement, September, 1939, p. 180-25):

> Useful mixer data have been misinterpreted as a result of an error in elementary arithmetic,
>
> The concrete was overmixed due to an erroneous criterion for mixing,
>
> The tests as regularly carried out at Grand Coulee Dam during the past 4 yr. have accomplished little more than to determine 12 or 24 times daily the errors inherent in the test method,
>
> Messrs. Patch and Ruettgers now wish to add six new criteria of mixer performance to the one originally announced,
>
> Practically every statement made by Messrs. Patch and Ruettgers in defense of the U. S. Bureau of Reclamation mixer test is contradicted by the tested submitted [by them],
>
> The application of this test at Grand Coulee has already caused the Government and the contractors to spend hundreds of thousands of dollars needlessly,
>
> A sand-cement was unintentionally used in the lower portion of Grand Coulee Dam due to overmixing, as a result of a misinterpretation of mixer data.

I then maintained that the concrete was overmixed at 2 min., the mixing time referred to by Mr. Patch, and that 1 min. would have been sufficient. The 16-line closure of Mr. Patch in the A.C.I. *Journal* may be epitomized by this quotation:

"I gave Mr. Abrams the benefit of assumed sincerity in the first discussion, and went to considerable trouble to clarify the purpose and soundness of the procedure, all of which data seem to have been misconstrued or ignored by Mr. Abrams, leaving the whole argument again pivoted on his own unsupported assumptions. I see no further value to anyone in continuing the discussion."

This statement was made by Mr. Patch in spite of the fact that my discussions filled 29 pages in the A.C.I. *Journal*, analyzed every value given and repeatedly requested more data in addition to the two tests that I was then able to find.

Here the matter rested for 4 yr.; in the meantime the mixer test had undergone considerable change:

1941, January. U.S.B.R. *Manual* gave two methods; Method A, the original Patch test; and Method B, based on the water-fines ratio that was suggested in my discussion,

1942, October. U.S.B.R. *Manual* dropped both Methods A and B and for the first time gave the pycnometer (specific gravity) method.

The paper tells us:

"Since the Bureau of Reclamation at the time had three other large dams under way on which the method was being used, arrangements were made for a systematic series of experiments to clarify the issues."

Contrary to Mr. Patch, others in U.S.B.R. did see "further value . . . in continuing the discussion."

PATCH TEST DISCARDED IN 1941

The paper states with reference to the Patch test (p. 1013):

"While the absolute accuracy of such determinations is low on account of assumptions of specific gravities of materials and amount of fine sand in the mortar which may change both within the batch and from batch to batch, the relative values are generally all that are required."

My discussion of September, 1939, showed that instead of "absolute accuracy":

"Mr. Patch computes 'original' weights of cement, sand, and water (as they were termed in the January A.C.I. *Journal*) which are known to be wrong. As a general principle it is not sound engineering to divide one wrong number by another wrong number and expect to get a right answer; this is what was done in computing sand-cement and water-cement ratios. The indication of these wrong values is then greatly exaggerated by computing a spread on the basis of the lowest, giving no weight whatever to the middle value or to the average."

I pointed out: (*a*) The test does not determine the weights of cement, sand, water, (*b*) The method was invalid on account of the necessity of assuming the quantity of 0–100 sand accurate to four significant figures, since the quantity of this sand actually varied from hour to hour and from batch to batch. Also that we could assume fine sand, passing No. 100 sieve, to vary from 20 per cent to −10 per cent, or the sand to have any specific gravity from 2 to 10,000 and it made not the slightest difference in the results as computed by Patch.

Both Patch and Ruettgers vigorously denied measurable variations in 0–100 sand, although the data they submitted showed a 50 per cent spread in adjacent shift averages of four samples and a 75 per cent spread in shift average in a few hours. The present authors admit the truth of my conclusions, but at the same time they have nothing but commendation for the discarded Patch method.

The Patch test was continued at Grand Coulee until the dam was completed in 1941, when it was discarded, but the paper goes so far as to introduce 2 "improvements." In Fig. 4 average water-cement and sand-cement ratios are used; my earlier discussions pointed out that a serious weakness of the Patch method was that he made no use of averages. In conclusion 2 the authors use "control limits of 15 per cent for water-cement ratio, 21 per cent for sand-cement ratio" as comparable bases of comparison in the "Patch method." The numerous papers by Mr. Patch, U.S.B.R. specifications for large concrete dams (Grand Coulee 1937, Shasta 1938, Friant 1939) and the "Concrete Manual" (1938, 1939, 1941) invariably used 10 per cent as the control limit for both these factors. The present use of widely different limits is an admission that the 10 per cent used for both ratios on the world's largest concrete dams, was not on a comparable basis. Why was the Patch test discarded for one that is "about 80 per cent as efficient"? Why is the discarded Patch test still used as a measure of the performance of a new test that is "adequate and practical"?

It should be reiterated that I have no quarrel with the *data* secured in the Patch test; they give a proper measure of mixer efficiency, but they were improperly interpreted by the U. S. Bureau of Reclamation.

THE NEW TEST

In order to carry out the new unit weight test we must go to the 1942 "Manual" where seven pages were devoted to it. Nearly every reference to the "new test" drags the now discarded Patch test along "by the hair," although in most features the methods are incompatible. We read (p. 1002):

"The new test cannot be expected to replace earlier methods of wet batch analyses where complete information concerning the distribution of each of the materials entering into a batch is required."

The new test actually did replace the Patch test as a U.S.B.R. "Standard"; see 1942 "Manual." There were 3 references to "wet batch analysis," the first (and obviously the preferred method) being the original Patch paper in the A.C.I. *Journal*, 1939. But:

(*a*) The Patch test is not a wet-batch analysis,

(*b*) It does not give "complete information."

Only two advantages of the new test were mentioned (p. 1001):

(1) ". . . substantial saving in time,"

(2) ". . . possible to express results in readily interpretable terms. . . ."

The saving in time is unimportant; and of course one measure of mixer efficiency can be expressed in "readily interpretable terms" just as well as another.

Suppose we assume three samples of 140.6, 140.9, and 138.5 lb., average 140.0 lb., range 2.4 lb., mean variation 1.00 lb. The possible range in unit weight is 193 − 63 = 130 lb. The mean variation of three samples is 0.8 per cent of the total possible range. According to any usual standard a mean variation of 0.8 per cent is small enough in dealing with concrete. The chances are extremely high if 5, 10, or 18 samples were taken the mean variation would be much less than 0.8 per cent (the authors did not try this with the 18 samples in Fig. 6) yet U.S.B.R. would reject the above batch, since the range of weights is 2.4 lb., whereas their limit is 2.3.

We find another conflict (p. 1003):

"After screening the samples through a $\frac{1}{4}$-in. sieve, they are weighed in air, and the weight of water displaced by each sample is determined either by the pycnometer method or by the immersion method such as used in the Patch test. The sample is stirred or agitated to free all entrapped air."

In the Patch method the sample was not "stirred or agitated."

It is difficult to determine the motives in dragging in the discarded Patch test, where it is not applicable, and making statements concerning it that are unwarranted and misleading.

Other phases of the new test are discussed below.

VARIABILITY COEFFICIENTS

The 1942 "Manual" (p. 434) gave a sample computation for what I infer is the "new test"; it is dated Nov. 8, 1938, and was obviously intended to indicate Grand Coulee Dam as the locale although the method had not been developed at that time, nor was it ever used in routine tests at Grand Coulee. The three samples, designated as front, center, back (of mixer) had the following unit weights: 144.3, 141.6, 145.0 lb.; also "air-free unit weight variability 145.0–141.6 = 3.4 lb." This test is the fourth instance that I have found in which U.S.B.R. gave actual data of tests; although they must have made 100,000 such tests.

I show below that "air-free unit weight variability" is no measure of perfection of mixing concrete, but for the present let us accept the above weights as significant. Neither the refinement of the method nor the number of tests justify complex statistical methods. Mean variation has long been used and is well adapted to this purpose. The average is 143.6, the mean variation = 1.4 per cent. My discussion of June, 1939, gave the mean variation of two earlier tests when correctly interpreted. The mean variation of a similar test in the 1941 Manual is 1.0 per cent. The mean variation of the four groups of three samples now available are:

Data from	Range by U. S. Bureau of Reclamation	Mean Variation of Three Samples by Abrams, per cent
U. S. Bureau of Reclamation "Manual" 1938	5.0% of W/C	0.6
Patch, Journal, A.C.I. 1939	6.0% of W/C	1.4
U. S. Bureau of Reclamation "Manual" 1941	17.1% of W/C	1.0
U. S. Bureau of Reclamation "Manual" 1942	3.4 lb. unit weight	1.4
Average		1.1

A correct interpretation of these tests (the only ones released) shows a striking uniformity of four mixes over a term of 5 yr., as indicated by mean variation of 0.6 to 1.4 per cent. The new test (as judged by one example) gave a mean variation of the same order of magnitude as the data from the Patch test. I maintain that mean variation of 0.6 to 1.4 per cent represents a more accurate control of concrete mixing than any one has a right to demand, especially when compression tests of concrete at Grand Coulee Dam showed ranges of 50 to 70 per cent between low and high values during one month, and tests in Fig. 3 of the paper, over an undisclosed period, show ranges of 43 and 56 per cent.

Where U.S.B.R. obtained a "variability index" of 17.1 per cent, my analysis shows mean variation = 1.0 per cent. It is notable that this extremely high error appeared *after I had criticized the Patch test* in 1939. The 1942 "Manual" value shows a range of 3.4 lb., which is higher than their limit of 2.3 lb.; my figures show mean variation = 1.4 per cent. In this instance the "Manual" stated:

"Mixing time, min., currently used 2.2
Apparently needed, min. .... 2.9"

Thus as late as 1942, U.S.B.R. advocated mixing 2.9 min. in order to reduce the range for a batch that had mean variation = 1.4 per cent. The illogical methods of computing mixer efficiency used by U.S.B.R. have always greatly exaggerated the small variations shown by their data.

REFUSAL TO DIVULGE SAMPLING METHODS

The significance of the sampling method may be seen from the fact that this question was raised in both of my previous discussions of the Patch test. Mr. Patch claimed that the mortar-mix test had enabled them to make important improvements in mixer design and operation at Grand Coulee Dam. My discussion stated:

"Improved mixing seems to have been coincident with a change in sampling; possibly the improvement resulting from taking out two thirds of the blades was related to this change."

Mr. Patch made a categorical denial, but neither he nor Mr. Ruettgers would divulge the sampling methods used in any tests made at Grand Coulee, although I called attention 15 different times to this omission.

The present paper reiterated the same claim (p. 1001):

"In Mr. Patch's hands the method proved adequate not only to determine when concrete was thoroughly mixed but also to determine needed improvements in the batching and mixing procedures,"

but still without having cleared up my original question of the influence of sampling methods. Five different U.S.B.R. writers in discussing mixer tests during the past four years have either refused to divulge or failed to give the methods of sampling.

In the tests in the paper two different methods of securing samples were used, neither of which was described. In the early tests at Grand Coulee "grab" samples were taken from the top of the batch after stopping the mixer. The essential features of grab samples are: (1) they are relatively small, (2) there is no blending with adjacent material. In 1936 grab-sampling was dropped and samples were taken by a new method which required the construction of a heavy swinging bracket attached to a support of the mixer building that was used to sweep a container through the stream of concrete during mixer discharge. These are entirely different from grab samples; they are not isolated, but represent a "ribbon" of mortar of considerable length, and consequently give a much more favorable and more accurate measure of mixer performance than grab samples.

The authors of the present paper attempt to slur over embarrassing questions by throwing the two methods of sampling together (p. 1003):

"The samples may be taken while the mixer is discharging, or from the top of the batch while the mixer is stopped."

In order to show complete impartiality we shall see that the two different methods were used in the same groups of tests. We shall see also some of the absurd results of taking grab samples from the top of a batch after stopping the mixer. In spite of the previous prolonged controversy on the relative merits of the two methods of sampling, the authors gave no results of tests in which both methods were used on the same batches.

TEST DATA NOT IN PAPER

This paper is characterized by the same lack of experimental data to which I called attention in my discussions of 1939. Half a column was devoted to "Test Data" from U. S. Bureau of Reclamation tests, which opens with this statement:

"The principal data of this paper are summarized in Fig. 1. . . ."

The data of the paper are the original experimental values. Figure 1 contains no test data, but consists solely of computed "variations" and "coefficients." The only values in the paper that could possibly qualify as "data" are 28 average unit weights in Figs. 4 and 5; these give no basis for computing mixer efficiencies.

The paper does not give one set of weights of air-free mortar, from which an independent computation of variation in mixer performance may be made. The next most notable omission is the absence of data on slump at different mixing times, in spite of the fact that my previous discussions had emphasized the vital importance of this information. In Figs. 1 and 6 slumps are given, but they are meaningless, since we are not told at what mixing time they were taken.

DISCUSSION OF FIGURES IN PAPER

This discussion will be restricted to U.S.B.R. tests. Attention will be called to many important omissions. Points on the figures will be accepted at face value, although discrepancies will be pointed out. I show later that a narrow range in weight of air-free mortar is no guarantee of perfect mixing.

Figure 1. Unit Weight versus Revo-

lutions of Mixer.—The following variables entered into the tests plotted:

Type of mixer,
Size of mixer,
Rpm. of mixer drum,
Number of revolutions,
Type, size and grading of aggregates,
Cement factor,
Time of admission of mixing water,
Water-cement ratio,
Slump,
Admixture replacing cement,
Method of sampling,
Number of batches.

With a dozen variables any one of which may affect the properties of mortar as determined by tests, it is readily seen that there is no common ground for comparisons between points plotted in Fig. 1.

One curve was based on two $2\frac{1}{2}$-cu. ft. laboratory batches. Here the surface area of the drum was so large as compared with the small volume of concrete that I do not believe these tests, using a $1\frac{1}{2}$-in. aggregate, should be given any weight whatever in a study that is principally concerned with 4-yd. mixers using 6-in. aggregates.

The entire upper portion of the figure to the left of 10 revolutions is based on 3 points which furnish the sole basis for the "semi-empirical" exponential equation represented by the heavy solid line. Even with this tenuous foundation the equation is given about five times as much space as was devoted to "Test Data." That the tests are inconclusive is shown by the fact that two mixers of the same capacity (4 yd.) showed "variations in air-free unit weight" at 5 rev. of 6 lb. for dam A and 10 lb. for dam C. Although seven batches were tested, there is a spread of 67 per cent between the performance of two 4-yd. mixers.

The "data" on tests covered by this figure are in important respects contradictory, incomplete and/or erroneous. I identify two of the "large dams" as:

Dam A	Friant
Dam B	Grand Coulee

Grand Coulee is referred to also (Fig. 4) as "a fourth dam."

At Friant the mixing conditions were probably the most severe encountered on any of these jobs; 21 per cent of the cement was replaced by a pumicite that had a higher specific surface than cement, thus adding an entirely different constituent that must be incorporated into the mix. The maximum size of aggregate was $8\frac{1}{2}$ in. instead of 6 as stated on Fig. 1. The 1-in. slump was much drier than the 3-in. used at Grand Coulee. All three of these factors, none of which are mentioned by the authors, are different from Grand Coulee Dam and make this concrete more difficult to mix. Apparently no sieve analysis of Friant sand has been published; this might have an important influence on these tests.

Since this investigation grew out of my criticisms of Grand Coulee practice (dam B), let us see what was found for this job. We are not told how the 20 batches for each of the five mixing times were chosen. With a high-power reading glass we can distinguish the open diamond-shaped points at 10, 15, 18, 22, 25 revolutions, but the authors nevertheless continued the thin-dash line representing Grand Coulee tests by extending it at both ends to the light squares, the symbol for dam C.

The Grand Coulee tests followed an entirely different pattern in that the samples were taken during the discharge of the mixer, whereas for dams A, C, and D grab samples were taken after stopping the mixers. The full significance of this difference in sampling methods will be seen under Fig. 5. There is ample evidence that the upper branches of these curves are due to: (1) an erroneous method of sampling, (2) sand gradings that were not adapted to a short mixing time, (3) mixing water admitted long after mixing began.

It is notable that for Grand Coulee (dam B) no tests were made at less than 10 revolutions although *10 revolutions was the very mixing period that my studies had shown to be sufficient.* No tests were made over 25 revolutions, although mixing up to 150 revolutions was actually required on the job. This job contributed nothing to the upper branch of the curve.

The ranges in unit weights for dams A and B are:

Revolutions of Mixer	Friant (Dam A) Slump, 1 in.	Grand Coulee (Dam B) Slump, 3 in.
10	3.0	1.7
15	1.7	0.8
20	1.3	0.7
25	1.3	0.6
Avg.	1.8	0.9

A curve plotted from the table under "Fig. 6" below shows that at 10 revolutions the 1-in. slump used in dam A would show a range of about 5 lb. as compared to 2.1 lb. for the 3-in. slump at dam B. The fact that Friant Dam showed a range double that of Grand Coulee should be borne in mind in our later comparisons. This difference is due to three causes: (1) pumicite at Friant, (2) lower slump at Friant, (3) difference in sampling methods. A range of 1.7 lb. at Grand Coulee shows that this concrete was well-mixed at 10 revolutions on the basis of U.S.B.R. limit of 2.3 lb.

Figure 2. Range Forms a Smooth Curve.—Important data omitted: origin of points on water-cement ratio curve; were unit weights for 5, 10, 15 revolutions higher or lower than at 25 revolutions?

Here the Friant (dam A) tests were lifted out of Fig. 1 for greater emphasis. The unit-weight curves, based on a mix with 21 per cent pumicite and 1-in. slump, gives a false impression of the mixing time required for the Grand Coulee mix with a slump of 3 in.

The "true variability" both here and in Fig. 1, is on a questionable basis. Figure 2 shows the following peculiarities:

Revolutions of Mixer	Experimental Error	
	Weight, lb.	Water-Cement Ratio, per cent
5	0	0
10	0.1	0
15	0.3	1
20	0.6	2
30	0.8	3
60	1.1	4

The authors should explain why there was an error of 1.1 lb. at 60 revolutions, and zero error at 5 and 10 revolutions; similarly for the water-cement ratio curve. I would expect greater errors at the short mixing times. If the "true variability" curve were drawn with a constant error of 1.1 lb. (the equation of the curve shows constant error), it would indicate that the concrete at dam A was satisfactorily mixed at 10 revolutions (1 min.) according to the U.S.B.R. criterion of 2.3 lb. range as a maximum. If this concrete (with pumicite, low slump, etc.) was well mixed, the concrete at Grand Coulee was sufficiently mixed in 1 min., as I pointed out four years ago.

The authors should explain how the water-cement ratio values in Fig. 2 were obtained. How distinguish pumicite from cement? It will be shown below that the correlation between *range* in water-cement ratio and *range* in unit weight should not be construed as a correlation between water-cement ratio and unit weight.

Figure 3. Correlation Between Strength and Unit Weight.—Important data omitted: slump; time covered by tests; age and number of compression tests for each point.

A tedious process of elimination enables us to conclude that these tests were made at Grand Coulee and that they represent some of the batches shown for 15 and 25 revolutions in Fig. 1. From other records we can supply missing data on type and size of mixer, type, size and grading of aggregate.

The compression tests show the following characteristics, as scaled from the diagram:

	1½ min.	2½ min.
Number of tests........	24	22
Maximum strength, psi..	4640	4550
Minimum strength, psi..	2980	3170
Average strength, psi....	3720	3920
Mean variation, per cent.	7.5	6.8
Range, low to high, per cent................	56	43

Regardless of the statistical drapery that is thrown around these tests it is extremely doubtful whether the increase of 200 psi. in strength between 1½ and 2½ min. was real. In the absence of the slumps it is impossible to answer the question definitely with reference to these tests; however, my earlier discussion showed the fallacy of a similar series of tests in Fig. 1 of the discussion of Mr. Patch (*Journal*, A.C.I., June, 1939), where slumps and strengths were reported for 6-in. aggregates mixed at Grand Coulee Dam for 1, 2, 4, 10, and 20 min. An increase in strength was shown, but this was entirely offset by a reduction in slump from 4½ in. at 2 min. to 1¼ in. at 20 min. My conclusion was that:

". . . 2 min. mixing with the water-cement ratio required for a 1¼-in. slump would have produced the same *strength* and *slump* as that actually found after 20 min. mixing using the water-cement ratio necessary to produce a 1¼-in. slump at that time.

"The writer fails to find in this test any evidence of 'improvements beyond the 2-min. mixing period in both compressive strength and slump' claimed by Mr. Patch."

Tests in Fig. 3 were obviously intended to be the response to the above, in a second attempt to show that long mixing of concrete resulted in an increase in strength. The authors did not give slumps, hence the tests in Fig. 3 cannot be accepted as evidence of increase in strength with mixing time. Suppose we assume a loss of slump of 1 in. from 15 to 25 revolutions. Information in the *Engineering News-Record*, Oct. 15, 1936, showed that at Grand Coulee increasing slump 1 in. required the addition of 40 lb. of water to a 4-yd. batch. There was 1505 lb. of cement, hence the loss of 1 in. in slump can be compensated only by the addition of $40/1505 = 0.027$ to the water-cement ratio of the batch. Since the original water-cement ratio was 0.54, this means an increase of 5.0 per cent in mixing water, and about the same *reduction* in strength. The increase in compressive strength shown above was $200/3720 = 5.4$ per cent. In other words, a loss of 1 in. in slump from 15 to 25 revolutions would almost exactly counteract the apparent gain of 200 psi. in strength, so that the apparent gain in strength with mixing time was probably imaginary.

It is important to note that the increase of about 2 lb. in unit weight from 1½ to 2½ min. mixing in Fig. 3 is directly contradicted by the *loss in unit weight* in Fig. 5. Difference in time of admission of mixing water and sampling methods may account for this.

We find an unwarranted statement concerning Fig. 3 (p. 1006):

"From the shape of the computed curves in Fig. 2 it is evident that range in unit weight is correlated with range in water-cement ratio and consequently with strength. The correlation of strength with average unit weight is shown in Fig. 3."

This shifts the blame for 56 per cent range in strength from variation in tests and quality of cement, as claimed by Mr. Patch, to "range in water-cement ratio." There is no evidence whatever to show that the wide scattering of points in Fig. 3 was due to range in water-cement ratio. It will be shown below under "The Unit Weight Test" that in general there is no correlation between water-cement ratio and unit weight of mortar. No correlation has been shown between the water-cement ratio of 2-lb. mortar and 30-lb. concrete samples.

Figure 4. Lack of Uniformity from Batch to Batch.—Not included in outline on p. 1003. Important data omitted: moisture content of sand at 2-hr. intervals; original weights in Patch test; slump; date of tests.

The evidence shows that these tests were made at Grand Coulee Dam and that the mixing time was 2½ min. Careless readers might conclude that these tests were lifted at random out of 200 weeks' operation of eight different mixers and were representative of Grand Coulee practice; but the authors make no such claim. They say only, "control charts show lack of uniformity from batch to batch," which anyone can see.

These results should be contrasted with statements of Mr. Patch who justified the reduction of mixing time for these mixers from the specified 2½ min. to 2¼ min. in 1936; to 2 min. in 1937, and to about 1¼ min. for a time in 1939. (***Engineering News-Record***, June 8, 1939, p. 749). At Grand Coulee concrete was not regularly mixed 2½ min. after 1936. In mixing this concrete for 2½ min. the authors revived a practice that had been discontinued 4 or 5 yr. before the tests were made.

The paper goes to great length to prove that the "control chart" was "unusual," for example (p. 1006):

"The chart for average unit weight indicates definite lack of control. Investigation of the data for the month from the job indicated that weighing of the separate ingredients was well controlled but that moisture content of the sand fluctuated enough that a maximum change of 5 lb. in unit weight could have been obtained. Evidently part but not all of this variation was controlled at the mixer by changing the added water to obtain uniform consistency."

This cryptic language calls for comment. It shows that the authors had no definite information on either the quantities or control of these batches. How can worthwhile conclusions be drawn from the tests when the authors admit that they guessed at both the quantity of mixing water and the time of its admission to the mixer, and then go on to speculate on the consistency of the concrete? What is the significance of "could have been obtained?" One might ask, what *was* obtained? Why "the month" when only a week was involved? How "obtain uniform consistency" when "part but not all of this variation was controlled?"

If the slump was "controlled at the mixer by changing the added water," it is quite obvious that during most of the 25 revolutions the batch was drier than normal, hence more difficult to mix. This was not a normal practice at Grand Coulee. When each mixer in a battery of four turns out a batch each 3¼ to 2¼ min., the operator has neither time nor inclination to "add water to obtain a uniform consistency."

Does the "change of 5 lb. in unit weight" refer to the sand or the mortar? If it refers to sand, it can be said definitely that under normal conditions no such fluctuation occurred at Grand Coulee. Moisture in sand was at all times under rigid control; a moisture determination was made each 2 hr. U.S.B.R.

"Manual" 1938, 1939, p. 124, reproduced the report from one shift in September, 1937, which showed five samples at 2-hr. intervals; moisture varied from 8.4 to 9.3 per cent.

U.S.B.R. "Manual" 1939 (p. 200) states:

"Also, the importance of having stable moisture conditions in the aggregates is illustrated by the fact that for say a 1:2.5:4.0 mix a 1 per cent change in surface moisture in the sand alone will change the slump about 1½ in."

Here the authors are speculating on a 7½ in. change in slump.

If the "change of 5 lb." refers to the mortar, it presents an incredible situation, since water is both the lightest and the smallest ingredient in the mortar. Using the mix given in Fig. 4 we find that the actual quantity of water must be changed about 30 per cent in order to change the weight of mortar 5 lb. per cu. ft. or from water-cement ratio of 0.47 to 0.61.

The reader is allowed to infer that if the batches show such lack of uniformity after 2½ min., they must be hopeless at 1 min., but the tests in Fig. 1 do not bear this out; they show dam B (Grand Coulee) to give a range of 0.6 lb. at 2½ min., whereas in Fig. 4 the average range was 2.04 lb.—3½ times as much.

We have noted three important departures from normal practice at Grand Coulee: (*a*) 2½ min. mixing, (*b*) uncontrolled moisture in sand, (*c*) "changing the added water." There may have been other departures. Slumps were not reported, hence the concrete may have been much drier than normal.

How can these "unusual" tests possibly help to "clarify the issues" on the very job in question?

Figure 5. Properties of Air-Free Mortar.—Important data omitted: sand grading; type of mixer; time of adding mixing water; individual weights in tests for water, cement, sand.

Two thirds of the curves in Fig. 5 were computed or estimated, hence are of little interest in this discussion. The heavy curves are based on seven batches from dam A and show:

". . . in a fundamental way what is actually taking place in the batch."

Weights at the bottom of the batch were "estimated" from them. We are justified in concluding that these are representative curves and indicate the considered views of U.S.B.R. It is extremely odd, that on stopping the mixer *heavier-than-normal air-free mortar was always found on the top of the batch;* in no case was lighter-than-normal mortar found on top. From the laws of probability, the chance of such a combination is so small as to be negligible. This should be compared with the "data" of Mr. Patch who showed that during a sample run of a week in November, 1938, at Grand Coulee Dam the mixers were *much more efficient at night than during the day.*

Figure 5 purports to give "Properties of Air-Free Mortar," but it is obvious that something is missing as shown below:

Mortar Constituent	Percentages from Fig. 5			
	Beginning	5 rev.	20 rev.	60 rev.
Water	14	11	12	12
Cement	20	13	18	18
Sand	67	71	65	65
Total	95	95	95	95

In every instance the total is 5 per cent short. This shows a fundamental error. Probably the authors forgot 21 per cent of pumicite. (*Reclamation Era*, March, 1941, p. 69.)

The authors do not comment on the above feature, but they gave two "probable" explanations of the "gradual decrease from a unit weight of 144 lb. at 5 revolutions to 142 lb. at 60 revolutions":

"First, grinding of coarse aggregate produces more sand which increases the quantity of high specific gravity material in the mortar."

This explanation contains two fundamental errors and an oversight:

With 6-in. aggregate the bulk of the grinding comes from pulverizing the sand,

According to my method of computation increasing "the quantity of high specific gravity material in the mortar" would increase its unit weight, rather than decrease it,

Again forgot to mention that 21 per cent of pumicite was used.

The other factor "probably responsible" was:

"Second, in separating mortar from concrete by wet screening, some paste (water and cement) is left on the coarse aggregate whereas practically all the sand is removed."

Of course "some" and "practically all" are quite indefinite, but it is not clear why cement was left on the coarse aggregate and sand of the same fineness removed, or why the amount of paste was not determined. Here again nothing was said of the pumicite. At any rate, the explanation is an admission that the Patch test does not give "complete information concerning the distribution of each of the materials entering into a batch," as stated on p. 1002; it did not reveal the presence of pumicite.

There are obvious explanations. The presence of 21 per cent pumicite replacing the same weight of cement, plus the 1-in. slump and 8½-in. aggregates made mixing more difficult. The water that we would expect to find at the surface of the batch settled as soon as the mixer stopped. Before sampling was completed this mortar was drier than normal, and consequently gave a higher-than-normal unit weight. Another probable cause is the addition of mixing water during mixing; still another is the evaporation of water from the early samples.

These phenomena probably account for most of the wide ranges that are found in unit-weight tests, where grab samples were taken by stopping the mixer during the first few revolutions. There are three obvious conclusions: (1) a scientific method of sampling is needed; (2) no reliance can be placed on the wide ranges indicated by grab samples taken during the first few revolutions; (3) for research purposes in studying short-time mixer performance all mixing water must be in the drum before mixing starts.

Three other comments may be made on Fig. 5: There is no basis whatever in the data for the curves marked "Estimated at bottom" of batch; they are absurd and give a false impression. All data from grab samples taken after stopping the mixer should be re-examined in the light of these disclosures. These tests made on Friant Dam do nothing toward clarifying the issues with reference to overmixing of concrete at Grand Coulee Dam.

Figure 6. Range in Unit Weight for Different Mixes.—Important data omitted: type of mixer; type, size and grading of aggregates; individual unit weights; were water-cement ratios determined by test or assumed? When was slump test made and what was slump at other mixing time?

This "regular mix" was not "regular" anywhere except on Friant Dam; some of its properties were discussed under Fig. 5. It is not clear why this freak mix was used in studies to "clarify the issues" on concrete mixing at Grand Coulee Dam. The paper states (p. 1009):

"In the two graphs at the top, the range of six samples is converted to an equivalent range for a set of three."

Test values were suppressed and statistical theory substituted. Statistical theory has its place; it may give significant indications when dealing with a large number of samples, but there is little assurance that it applies to a single sample lifted at random out of a "universe."

The authors do not give us ratings on these five batches, but:

"Ranges of unit weight for sets of three are plotted on the diagrams and will repay study."

Original weights would furnish a more profitable basis for study. According to U. S. Bureau of Reclamation criterion there are 816 different ranges in 18 samples. Which one would they use? The actual *variation*, the only true measure, could be computed in a few minutes. Apparently the authors did not do this.

Let us confine ourselves to the samples adjacent to the mixer axis, this conforms to the "front, center, back" formula for securing three samples. The average ranges of two sets of samples at 10 revolutions were:

Mix	Cement		Slump, in.	Range at 10 rev., lb.	Estimated Mean Variation at 10 rev., per cent
	U.S.B.R.	Actual			
SLUMP UNIFORM, CEMENT VARIED					
Lean	0.51	0.65[a]	1¼	5.2	2.1
Regular	0.79	1.00[a]	1¾	3.3	1.4
Rich	1.38	1.75[a]	2	0.5	0.2
CEMENT UNIFORM, SLUMP VARIED					
Dry	0.80	1.00[a]	¾	6.0	2.5
Regular	0.79	1.00[a]	1¾	3.3	1.4
Wet	0.78	1.00[a]	5	1.7	0.7
ESTIMATED RANGE FOR GRAND COULEE					
	1.00	1.00	3	2.1	0.9

[a] Includes pumicite.

If we plot a smooth curve of slump *versus* range for the "cement uniform" we can take off the range for 3 in. slump used at Grand Coulee as 2.1 lb.; since this is a single batch it conforms to U.S.B.R. maximum limit of 2.3 lb. This does not take account of the fact that the 2.1 lb. was based on the presence of 21 per cent pumicite.

There is an entirely independent approach to this problem. We saw above that three tests of unit weight of air-free mortar in the 1942 "Manual" gave a range of 3.4 lb. and mean variation of 1.4 per cent; in other words a range of 2.4 lb. represents a mean variation of 1 per cent. If we apply this factor to the range given above, we secure the mean variation in the last column of the table, which indicates a mean variation of 0.9 per cent for the Grand Coulee mix at 10 revolutions, a low enough error in concrete mixing. In fact this shows a lower mean variation than we found above under "Variability Coefficients" for tests actually made at Grand Coulee but mixed for 20 to 25 revolutions.

These tests confirm in two different ways the conclusion in my discussion of the original Patch paper and repeatedly shown by later tests submitted by him, namely that Grand Coulee concrete was sufficiently mixed at 1 min. (10 revolutions).

The above tables exhibit a fundamental weakness of the unit weight method. It is not reasonable that this mixer at 10 revolutions is 10 times more efficient with the rich mix than with a lean one when practically the only difference between them is 4 bbl. of cement, which we would suppose to be the most difficult material to mix. The difference probably can be traced to unscientific sampling. This case is similar to what we saw in Fig. 5. When the mixer was stopped for sampling, the rich mix held the water, hence wide ranges in unit weights were not found.

The authors state: "At 10 revolutions the mortar is more variable at the back of the batch. . . ." This cannot be construed as a general conclusion, but is probably a characteristic of the Bessemer-converter type of mixer, first used on Grand Coulee Dam 1935 to 1941.

Figure 7. Four Measures of Variability.—Tests not covered under "Test Data" (p. 1003). A dozen lines (p. 1011) were devoted to this figure. The internal evidence indicates that the compression tests were not made from the same batches or with the same mixer as the other tests. We only regret that the authors considered it desirable to suppress data on:

Type and capacity of mixers,
Cement factor,
Type, size and grading of aggregates,
Slump,
Methods of sampling,
Whether weights were above or below normal,
How water-cement ratio and sand-cement ratio were obtained,
Strength of concrete for different mixing times.

The conclusion: "four measures of variability have similar shapes," when applied to strength hinged on one point, that is, a range of 17 per cent at 10 revolutions. One point cannot dictate the form of a complex plane curve when all other points show a distinctly different pattern, unless we assume in advance what the curve is to be; if we do this, we are assuming the only thing that was to be determined, namely the form of the curve. If we disregard the 17 per cent point, the strength curve becomes a straight line with a slope, as determined by four points, directly opposite to that of the other "variability curves." The figure shows a lower strength range at 15 revolutions than at 21, 31, or 42 revolutions. If strength uniformity is the criterion of mixer efficiency, 15 revolutions (1½ min.) with a range of 6 per cent in strength, is the optimum mixing time. This is the eighth new criterion suggested. The diagram furnishes no basis for the conclusions drawn from it by the authors.

TESTS AT GRAND COULEE DAM

We can identify three groups of tests as having been made at Grand Coulee. One group (Fig. 4) embodied three unusual practices that make these tests misleading. Another group is contradicted by other tests in the paper: the increase in unit weight with mixing time shown in Fig. 3 is contradicted by Fig 5. In the third group, Fig. 1, the Grand Coulee tests covered such a narrow range that they were practically lost among the other coefficients.

1 MIN. MIXING AT GRAND COULEE DAM

We have seen that most of the tests made to "clarify the issues" on concrete mixing on Grand Coulee Dam were made at Friant Dam where the materials, slump, etc., were entirely different. One of my criticisms of Grand Coulee practice was that the concrete was overmixed at 2 to 15 min. and that it was well-mixed at 1 min. Figure 2, if corrections are made for slump and pumicite, shows the concrete to be well-mixed at 1 min. In Fig. 4 the tests were admittedly "unusual," hence cannot furnish any information. From Fig. 6 we determined by interpolation from a slump-range curve that concrete of the 3-in. slump used at Grand Coulee was well-mixed at 1 min. and by an independent approach we found a mean variation of 0.9 per cent at 1 min.

Every instance in the paper where comparable conditions were found indicates that the concrete at Grand Coulee was well-mixed at 1 min. (10 revolutions) according to the new criterion. This confirms the conclusions of my discussions of Patch, 1939.

SAND CEMENT IN GRAND COULEE DAM

A 8-ton batch of water, cement, sand, gravel up to 6 or 9 in. in diameter constitutes an ideal grinding machine. One of the important "issues" in my 1939 criticisms was that overmixing the concrete at Grand Coulee Dam produced a sand-cement as a result of grinding sand in the mixers. My analysis of the limited data showed that when mixing was continued to 15 min. fine sand was ground equal to the weight of cement in the batch.

Earlier U. S. Bureau of Reclamation writers on the significance of the mixer test persistently refused to discuss this phase of their work at Grand Coulee Dam. The authors show that they did not overlook it and were not afraid of mixer grinding, since it was mentioned three times:

". . . grinding of coarse aggregate produces more sand . . ." (p. 1009),

". . . tests by Waugh, who found after 30 revolutions, in four out of six tests, a small increase in total sand" (p. 1009),

". . . grinding increasing the specific surface of the cement" (p. 1012).

This gives the reader four distinct impressions: (1) it is problematical whether grinding occurs at all (in "four out of six tests a small increase"), (2) coarse aggregate is ground, (3) increases specific surface of cement, (4) nothing of this kind had been noticed at Grand Coulee or on other U.S.B.R. jobs.

The tests by Waugh made on Hiwassee Dam where grinding caused a breakdown of the coarse aggregates were not at all comparable; both fine and coarse aggregate consisted of a crushed graywacke that was so soft that much of it could be crumbled in the hands, whereas at Grand Coulee the coarse aggregate was a hard, rounded basalt, granite and rhyolite gravel.

Every student of this subject has found that under normal conditions sand is ground rather than coarse aggregate. This was the case at Grand Coulee, but it would lead us too far afield to present the evidence here.

A large quantity of rock dust is undesirable for the reason that it takes about the same quantity of mixing water to produce a plastic mix as does the cement, hence makes necessary a higher water-cement ratio; this reduces both the strength and durability of concrete. With a rich mix, that is a high cement factor, little rock dust is ground, due to the cushioning effect of the cement, but with the comparatively lean mix used at Grand Coulee, 1 cement to 2.5 sand by weight (later changed to 1 to 2.7 by weight; 1 to 3 by volume), the first tendency is to grind enough sand to form a cushion, after which grinding proceeds at a much lower rate.

There was ample evidence of mixer grinding at Grand Coulee; it was always shown by the Patch test; it was evident from an examination of the fresh concrete in the dam; other data show it. It is the purpose of this section to present a new study of mixer grinding at Grand Coulee Dam and to see how this issue was "clarified" by the paper.

Mixer Grinding from 2 to 15 min.—Mr. Patch (*Journal*, A.C.I., June, 1939, p. 180-17) gave a data on the changes in slump of a batch using 6-in. aggregates, sampled after mixing 1, 2, 4, 10, and 20 min. While these tests are incomplete they furnish the best measure we have of mixer grinding at Grand Coulee. At 2 min. mixing the slump was 4.6 in.; at 15 min. it was 2.2. This loss of 2.4 in. was due to mixer grinding. Data in *Engineering News-Record*, October 15, 1936, showed that 40 lb. of water must be added to a 4-yd. batch to increase slump 1 in. In order to increase the slump 2.4 in. we must add 96 lb. of water. We now must determine how much cement can be converted to a plastic mix by 96 lb. of water. From Fig. 6 we have:

	Regular	Rich	Difference
Cement, bbl. per cu. yd.	0.79	1.38	. . .
Cement plus pumicite, bbl. per cu. yd.	1.00	1.75	0.75
Slump, in.	1¾	2	. . .
Water, lb. per cu. yd.	202	229	. . .
Corrected to 1¾-in. slump	202	226	24

The water-factor of this cement plus pumicite may be determined by dividing the weight of water added by the weight of added cement, or 24/0.75 × 376 =

0.085. In other words each pound of cement required the addition of 0.085 lb. of mixing water in order to retain the same slump.

We shall not be far in error if we use the same water-factor for the rock dust ground in the mixer. The rock dust that must be ground to account for 96 lb. of water is 96/0.085 = 1130 lb. There was 1505 lb. of cement in a 4-yd. batch, hence we have rock dust equal to 1130/1505 = 75 per cent of the cement.

It will be of interest to compare the water-factor obtained above with that determined in my original studies of this subject in 1928, using a $3\frac{1}{2}$ cu. ft. mixer, $1\frac{1}{4}$-in. aggregate and entirely different cement, sand, gravel. Those studies[4] gave a water-factor at low slump of 0.11. With the difference in batch sizes, etc., this is a fair agreement.

Mixer Grinding During First 2 min.—We saw above that at Grand Coulee 75 per cent of rock dust was ground from 2 to 15 min. Those tests did not lend themselves to a determination of the grinding during the first 2 min. But U.S.B.R. has furnished data on that phase. The Patch test always gave a lower water-cement ratio than the weighed quantities called for, which means that something was added to the "cement" during mixing; this gives a measure of mixer grinding, but no data have been released except for 2 or $2\frac{1}{2}$ min. mixing. Available data may be arranged:

Data from	Mixing, min.	Water-Cement Ratio			
		Original, by Weight	After Mixing	Loss, per cent	Corrected to 2 min., per cent
Manual, 1938, 1939	$2\frac{1}{2}$	0.621	0.494	26	21
Journal, A.C.I., Jan., 1939	2	0.564	0.475	18	18
Avg.					20

The loss of water-cement ratio exactly measures the increase in "cement," since there was no change in mixing water. There was undoubtedly some grinding of cement, but there was an actual increase of 20 per cent in "cement," which can be attributed to no cause but fine sand and rock dust.

Total for 15 min.—The total amount of rock dust in 15 min. mixing was 75 + 20 = 95 per cent in terms of weight of cement. This confirms my conclusion of 4 yr. ago, arrived at in a different manner, that a sand-cement was used in the foundation of Grand Coulee Dam.

To Minimize Mixer Grinding.—If one *must mix $2\frac{1}{2}$ min.* as specified, or 5 to 15 min. as frequently required at Grand Coulee Dam, the mix proportions should be adjusted accordingly. The amount of rock dust would be greatly reduced by using a larger quantity of sand, with an excess of coarse material. Mixer grinding would expend itself on the coarse material in the sand and make up the deficiency in fines with little or no excess. Instead, the opposite course was pursued in every instance: practically all glacial clay was washed out, 0–100 sand was reduced by washing to 6 per cent, a comparatively fine sand was used (fineness modulus = 2.65), a minimum quantity of sand was used.

Nearly all cushioning material was washed out of the sand and the concrete was overmixed, thus producing a set-up that resulted in the maximum amount of rock dust. A sand of any desired size or grading could have been used, since the sand was separated into four fractions of which three were recombined for the grading desired. Columbia River was practically dammed by clay and fine sand from the washing plant, and an additional 20,000,000 tons of sand was wasted over the mountain side.

If, say, 1 min. mixing had been used, a minimum quantity of a small-sized sand with a generous amount of glacial clay would have given optimum results. As it was the contractor at Grand Coulee Dam was penalized three times:

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[4] Duff A. Abrams, "Tests of Powdered Admixtures in Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 628 (1929).

1. Washing out glacial clay,
2. Wasting good sand,
3. Long-mixing that would have been unnecessary if more sand had been used and clay had been allowed to remain.

The presence of 95 per cent of fine sand and rock dust after 15 min. mixing at Grand Coulee Dam should be contrasted with the A.S.T.M. Tentative Specifications for Ready-Mixed Concrete (C 94 – 42 T)[5] and the Recommended Practice of the 1940 Report of the Joint Committee on Concrete and Reinforced Concrete[6] both of which permit concrete to be mixed 1½ hr. and "under favorable conditions" for 3 hr.

The paper completely concealed from the reader the sand-cement in Grand Coulee Dam which was an important "issue" of my previous discussions of the Patch test.

MIXER *versus* JOB SAMPLES

My discussion of September, 1939, stated (p. 180-29):

"The 4-yd. batches at Grand Coulee were turned over two or three times after the test samples were taken and before the concrete was in place; this has an important influence in improving the uniformity of the concrete in a given batch."

The response is found in the paper (p. 1011) where tests were interpreted as showing:

". . . a trend toward decrease in uniformity of the concrete with handling."

The brief view we are given of these tests show the following peculiar features:

The tests were not made at Grand Coulee.

We are not told the type, size or grading of aggregates, cement factor, slump, type or size of mixer, type of bucket, method of handling the concrete, or the method of securing test samples.

No explanation was given of the 10 to 12 per cent "decrease in uniformity of the concrete with handling."

It seems to me that this "trend" is rather alarming. The "data" may be arranged:

Number of Jobs	Time of Mixing, min.	Number of Tests	Range in Unit Weight, lb.		
			at Mixer	at Forms	Difference
2	2½	15	1.0	1.1	0.1
1	6	5	1.1	1.25	0.15
Average			1.05	1.175	0.125

U.S.B.R. rejects batches that show a range of over 2.3 lb. These batches had an average range of 1.05 when they left the mixer hence a gain of 1.35 would cause their rejection. This would occur if the batch were turned over 1.35/0.125 = 11 times as often as it was on these jobs.

But there is an obvious explanation. Methods of sampling were not given, but the method was probably shifted between the mixer and the forms. The mixer samples were probably taken by sweeping a container through the discharging concrete, thus a ribbon of mortar of considerable length was secured; whereas grab samples were taken on the job. Grab samples are the more variable by probably 30 to 50 per cent. A correct interpretation of the tests would show a *decided improvement in uniformity at the forms*. It is unreasonable that concrete of the slump used in a dam should become rather well mixed by being turned over ten times in a mixer and then become unmixed by being turned over two or three times outside a mixer. The authors compel me to speculate with reference to sampling methods, in spite of my repeated requests for this information.

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[5] 1942 Book of A.S.T.M. Standards, Part II, p. 1196.

[6] Report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete submitting "Recommended Practice and Standard Specifications for Concrete and Reinforced Concrete," Am. Soc. Testing Mats., June, 1940. (Issued as a separate publication.)

TIME OF MIXING CONCRETE

My discussion of the Patch paper 1939 (*Journal*, A.C.I., p. 180–1) stated with reference to mixing times as given in the U.S.B.R. Specifications for Grand Coulee Dam:

"There is no doubt of the right of the Bureau to demand that a 4-yd. batch be mixed $2\frac{1}{2}$ min.; however it should be stated that there is no rational basis for such a requirement or for increasing mixing time with increase in mixer capacity."

We find the response in the paper (p. 1011):

"It is not the purpose of this paper to specify the proper amount of mixing for any given job, but certain guiding principles may well be pointed out."

During the past dozen years the U.S.B.R. wrote into the specifications for large dams (Boulder, Grand Coulee, Parker, Marshall Ford, Shasta, and Friant) a definite requirement of minimum mixing times for mixers ranging from 2 to 6 cu. yd.; now they can only lay down "certain guiding principles." This is admission of the correctness of my conclusion, written 4 yr. ago. I was discussing a specification written in 1934; there is still no rational basis.

The authors now abandon *time of mixing* entirely and attempt to find a rational measure of mixer efficiency in the number of revolutions of the drum. We read (p. 1004):

"Analysis of wet ball mills of different diameters has shown that their performance *tends* to be similar when operated at the same percentage of the 'critical speed.' Concrete mixers are *ordinarily* operated at *about* equal percentages of critical speed, so it *seems reasonable to consider* mix variability as a function of mixer revolutions so that a direct comparison of the performance of the difference size mixers is possible. For mixers of equally efficient design, the time required to produce a given degree of uniformity will vary as the square root of the diameters, ..."

I italicized certain words in order to direct attention to them. After applying three different qualifying expressions, the authors quickly transformed a mere *tendency*, in the ball mill tests, into a positive assertion that in concrete mixers "uniformity *will* vary as the square root of the diameters"; this is restated in conclusion 3, quoted below. In the words of Mr. John Dewey (*Human Nature and Conduct*): "They converted a problem into a solution which afforded a simulated satisfaction."

The switch from mixing time to the number of revolutions was based on three qualifying expressions plus "it seems reasonable to consider." It does not seem reasonable to me for four reasons: (1) The function of a concrete mixer is entirely different from a ball mill; the former is designed to make a uniform blend of four or more materials of different characteristics, the latter to grind to a fine powder a previously prepared uniform feed. (2) The rpm. of mixers is the result of a guess by the manufacturers, who probably never heard of "critical speed." (3) The Bessemer-converter type of mixers used in these tests (See Fig. 6) were different from any heretofore used, hence there is no justification for general conclusions. (4) The *tends to be similar*, in the case of ball mills of different sizes was misapplied. What is the *performance* of a ball mill? The tendency was toward a linear relaship between the *useful power input* of the mill and its *capacity*, for a fixed grinding value. The authors gave no data on either the power input or the grinding characteristics of their mixers, hence there is no basis for comparison with ball mill practice.

In fact the authors are not consistent in their conclusion. They used a grinding machine (ball mill) as the basis for some of the most significant features of the paper, with reference to concrete mixing, but as we saw above under

"Sand-cement in Grand Coulee Dam," they refused to recognize concrete mixers as a grinding machine.

Conclusion 3 of the paper:

> "For a given uniformity and operating at the same percentage of critical speed, concrete mixers of different sizes require mixing periods directly proportional to the square roots of their diameters,"

is contradictory and speculative. "Directly proportional" signifies a linear relationship, hence we must choose between the diameter and its square root. It is speculative for the reasons given above plus the additional reason that there is no basis for this conclusion in the concrete tests submitted. No tests were reported in which mixers of similar design, but of different sizes, were used with the same materials and mixes; no information was given on "critical speeds."

Mr. Patch apparently was speculating also when, with the same mixers in mind, he stated an entirely different "law" (*Journal*, A.C.I., June, 1939, pp. 180–189):

> ". . . the length of mixing time required is roughly proportional to the diameter of the mixer drum."

Neither the data presented in the paper, nor those developed elsewhere, so far as I am informed, give a scientific basis for either of the similitude factors mentioned by U.S.B.R. writers for concrete mixers of different sizes. My studies of the meager *data* presented by U.S.B.R. writers indicate that concrete in a 4-cu. yd. machine is as well mixed at 1 min. as that in a $2\frac{1}{2}$-cu. ft. machine. Theoretical considerations suggest that, for a given way of charging, a well-designed mixer of large capacity would be more effective than a small one of the same pattern. If the correct similitude factor is ever found, it will be discovered from studies of concrete mixers rather than by any analogy with ball mill practice.

The authors have little faith in their own conclusion since they are unwilling to venture a recommendation on: mixing time for mixers of different size, the number of revolutions required, or the rpm. of the drum. If they are unwilling to do this after having placed more concrete than any other organization and spent probably $500,000 in testing and studying concrete mixers during the past 10 yr., where are we to get the answers?

VARIATIONS IN CONCRETE STRENGTH

My discussions of 1939 showed that at Grand Coulee Dam, U.S.B.R. insisted on mixer performance measured by a mean variation of about 1 per cent, while Mr. Patch did not even comment on a spread of 70 per cent in the strengths of compression cylinders from the same batches in a single month. We find a response to this criticism (p. 1011):

"Numerous series of tests show that if the range in unit weight within a batch is kept within an average of 0.6 to 0.8 lb., the corresponding ranges in strength will be about 5 per cent or have a coefficient of variation of about 3 per cent."

This important conclusion was based on off-stage tests; a range of 5 per cent is usually expected to give a coefficient of variation of about 1.6 per cent, a value much lower than any that has come to my attention, even in the most carefully controlled laboratory tests of concrete. Besides, the quote is directly contradicted by this statement in the Synopsis:

". . . a unit weight range of 1 lb. corresponds to a strength range of approximately 5 per cent for cylinders from the same batch. Applied to batch-to-batch control of concrete manufacture, test results indicate with 15 to 25 revolutions that concrete from batch to batch is 25 to 50 per cent more variable than that indicated within the batch."

There are other peculiar features of the last quote:

Batch-to-batch variation has nothing to do with mixer tests,

No such conclusion was stated in their paper on "Variations in the Strength of Portland Cements . . .," by Wing and Ruettgers,

My analysis of the data presented by Wing and Ruettgers shows that instead of 25 to 50 per cent more, the batch-to-batch variation was 250 to 400 per cent more than the within-batch variation, and from the standpoint of *variance* 6 to 15 times as much.[6a]

FALLACIES OF THE UNIT WEIGHT TEST

The fallacies of the new test method were outlined at the beginning of this discussion. They are illustrated and demonstrated by the accompanying Fig. 1. We learned in our study of plane geometry that from any point on the boundary of or within an equilateral triangle the sum of the distances measured at right angles to the sides is constant and equal to the height of the triangle. This principle enables us to construct a diagram on which we may represent the composition of any three-component mixture.

If the altitude of our triangle is divided into 100 equal parts, each part represents 1 per cent of the weight of a given material. Each point in the triangle will represent a definite composition of the three-component mixture; conversely every possible composition will be represented by a point on the chart. In our application of the diagram we have mixtures of water, cement, and sand. We may deal with them as percentages of the total, in terms of specific gravities, or of air-free unit weights. The last method is more direct for our purpose; percentages by absolute volumes can be read from the diagram; specific gravities can be secured by dividing the weights shown by 62.4.

If we give water, cement, sand positions at the apexes of the triangle (any order may be used) each apex represents 100 per cent of one constituent, the sides of the triangle represent all possible combinations of the materials mixed two at a time; points inside the triangle represent all possible combinations of the three materials.

Water, sand, cement, were assumed to weigh 63, 163, 193 lb. per cu. ft.; this keeps the main divisions of the chart in whole numbers, and gives unit weights that for the usual range are in error not over 1 per cent, for sand having a specific gravity of 2.65 and cement 3.10. We can trace the locus of points of a given unit weight by drawing a straight line joining points on the sides that correspond to these weights. Parallel lines cutting across the triangle show all points of a given unit weight, at intervals of 5 lb. per cu. ft.

The diagram shows also the locus of points having given water-cement and sand-cement ratios. We can locate a mix on this chart, if we know two of the water, cement, sand, components; the third is known from the fact that the sum of the three is 100 per cent; or we can locate a mix if we know *any* two factors that are independent of each other; for example, unit weight and water-cement ratio, or unit weight and sand-cement ratio. The diagram clearly shows that unit weight alone *cannot define a given mix;* on the contrary each unit weight represents an infinite number of different mixes.

When we mix mortar (or mortar in concrete) we start with three components; with the slightest commingling the three points at the apexes of the triangle move to a single point within. Suppose we could secure a sample of mortar from a point in a concrete mixer 1 ft. within the batch from the lowest point in the mixer at its mid-length at the end of each revolution, and could quickly analyze the mix and determine the percentages of water, cement, and sand. If these values were plotted in order on the chart, they would no doubt show a zigzag path for the first five or six revolutions, then rapidly settle down to a single point, as nearly fixed as the refinement of the test permits. The history of samples taken at any other place in the mixer would be similar. This single point represents a *definite unit weight and a definite water-cement ratio;* no other point on the chart can represent this well-mixed mortar. This shows at once two fallacies of the paper; assuming that *unit weight alone* can define a well-mixed mortar; and *assuming that any unit weight will do.* Mortars of a given water-cement ratio may have many different unit weights; conversely mor-

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[6a] See p. 946.

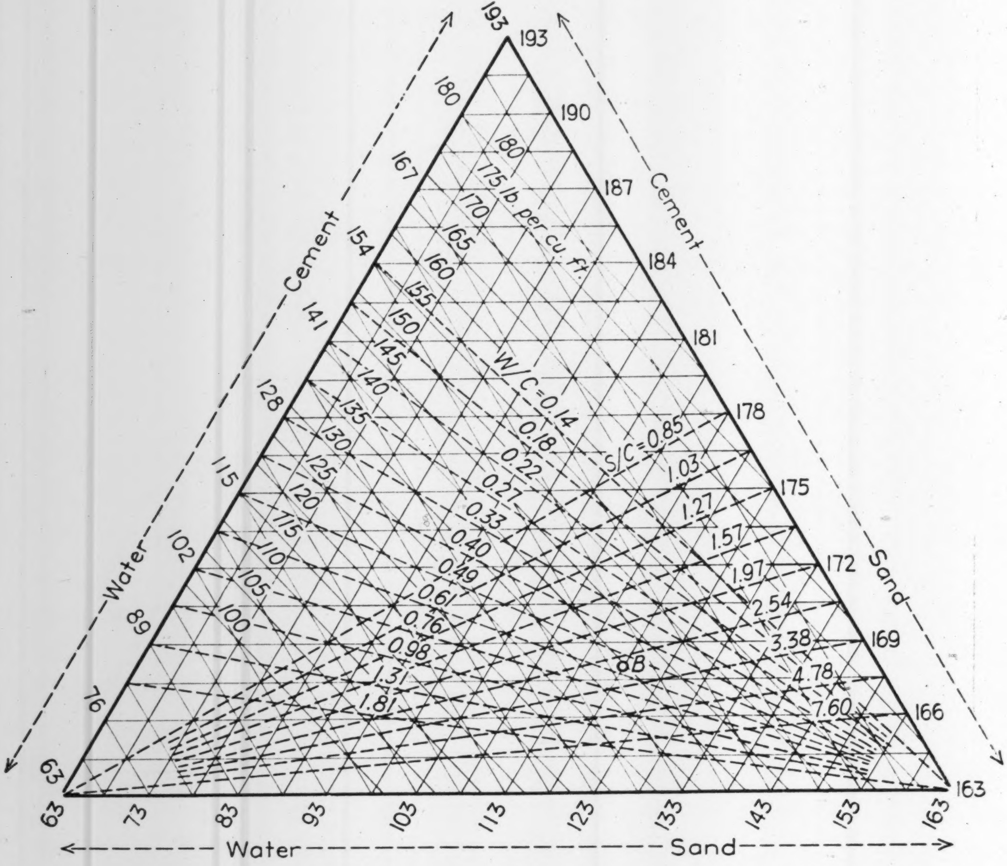

FIG. 1.—Unit Weight of Mortar.

Figures on boundaries are air-free weights of water-cement, cement-sand, and water-sand mixes.
w/c = water-cement ratio, by weight.
s/c = sand-cement ratio, by weight.
Assumed solid weight:

Water............................ 63 lb. per cu. ft.
Sand............................ 163 lb. per cu. ft.
Cement.......................... 193 lb. per cu. ft.

tars of a given weight may have many water-cement ratios.

Suppose the true point is at B on the chart—weight 140.0 lb. and water-cement ratio of 0.55. When the batch is well mixed, samples taken at other places will show this make-up. U.S.B.R. accepts a batch as well mixed if the points representing the three samples fall anywhere along the 140.0-lb. line, although at one extreme there is only water and cement, at the other water and sand. A batch that showed the following tests would be accepted as perfectly mixed:

Weight, lb.	Water-Cement Ratio	Sand-Cement Ratio
140.0.	0.33	0.85
140.0.	0.41	1.57
140.0.	1.15	7.60

The range in unit weight is zero, but a glance shows that the mortar is not mixed. This is only a part of the error since the method accepts as well mixed a batch three samples of which fall on (or near) *any other unit-weight line.*

35,000,000,000 MIXES OF ZERO RANGE IN UNIT WEIGHT

Suppose the mortar has a unit weight 145.0 lb. and a water-cement ratio of 0.61 when perfectly mixed. Strictly, there is an infinite number of mixes that may have this weight, but limitations of the test enable us to reduce this to a finite number. If the quantities of water, cement, sand were determined to the nearest 0.1 per cent, there will be 100 "stations" between each of the divisions of the accompanying Fig. 1. The length of the lines in the middle of this zone is 8.4 small spaces; the average number of "stations" will be 840. Any three points will do, hence the different mixes is the number of combinations of 840 things taken three at a time, or 840 × 839 × 838/6 = 100,000,000. All of these would be accepted as perfectly mixed under U.S.B.R. criterion, but only one is perfectly mixed, namely the one represented by three samples *each of which* weighs 145.0 lb. *and* has a water-cement ratio of 0.61.

The above were found on the 145.0 line, but there are many similar lines. The authors define no limits but recognize a range of 25 lb. Suppose we assume that weights between 130 and 165 lb. would be accepted. Unit weight determinations may be made to 0.1 lb., hence there are (165 − 130) × 10 = 350 different unit-weight lines. The total number of mixes within this zone will then be: 350 × 100,000,000 = 35,000,000,000.

It is a poor criterion of mixer efficiency that: (1) does not count a high range unless it is "substantiated by a check test," (2) arbitrarily disregards one half of all possible mortar mixes, (3) agrees in advance to accept any one of 35,000,000,000 mixes most of which are known to be not perfectly mixed.

THE UNIT WEIGHT TEST

The line for water-cement ratio = 0.33 bisects the diagram in Fig. 1 and represents about the low limit of air-free mortars, as they exist in concrete. U.S.B.R. method makes all mixes above this line air-free by adding water to the pycnometer. This, of course, creates an artificial mix which is not representative of what was taken from the mixer.

In order to show some of the other fallacies of the unit weight test we give three examples:

1. A unit weight of 140 lb. may have a water-cement ratio of any value above 0.23 and the strength would be changed accordingly,
2. A water-cement ratio of 0.61 may have any unit weight from 109 to 163 lb.,
3. As unit weight increases from 135 to 140 lb. its water-cement ratio may *increase* from 0.27 to 1.81.

Every statement in the paper that attempts to correlate water-cement ratio and unit weight is erroneous. There is no foundation either in the tests or in the fundamental relations involved for conclusion 4.

WANTED—DATA FROM MIXER TESTS

Spokesmen for U.S.B.R. have repeatedly refused to give the original values from which an independent computation of mixer efficiency can be made. Since the mixer tests are again brought to the fore, concrete engineers are now justified in demanding that the data be released. Specifically, I suggest the following:

From Grand Coulee Dam:

The original a, b, c, weights in the Patch tests, water-cement ratio, slump and 28-day concrete strength for:

(*a*) All batches mixed 5 to 15 min.,

(*b*) All mixers during the last 2 weeks of grab sampling,

(*c*) All mixers during the second and fourth weeks after the new method of sampling was adopted,

(*d*) All mixers for second week in April, May, June, July, August, September, October, and November, 1937,

All omitted data noted in my earlier discussions of Grand Coulee mixer tests.

Tests in Paper:

Fig. 1—A complete schedule of individual unit weights for all tests at dams A, B, C, D.

Fig. 2—Original a, b, c weights in water-cement ratio tests.

Fig. 4—Similar records for the other mixers for same week and for all mixers for third and fifth week before and third and fifth week after, including time of mixing, unit weights, original weights in Patch test, and moisture content of sand at 2-hr. intervals.

Fig. 5—Unit weights for each of seven batches.

Fig. 6—Unit weights for each of five mixes, in order of taking 18 samples.

All other missing data noted in this discussion.

No final evaluation of the concrete mixing blunders on Grand Coulee Dam can be made until these data are released. The routine test data from Grand Coulee will be adequate to "clarify the issues" that were raised in my discussions of 1939. A report on the above could be placed in the Engineering Societies Library, New York, where it would be available to all.

Principal conclusions were given in the Summary.

MESSRS. S. P. WING,[7] VALENS JONES,[7] AND R. E. KENNEDY[7] (*authors' closure by letter*).—The tests presented by the authors were primarily undertaken to determine the range of results which would be obtained in applying the Patch mixer test to several jobs with different operators, several types of concrete mixes, and systematic variation of mixing time. Data were desired on the reproducibility of results and on test errors, questions raised in a prior paper.[8] In opening their presentation the authors referred to this paper and its discussion as a reason for undertaking the test program. Mr. Abrams wrongly, but perhaps naturally, has assumed that the present paper is principally an attempt to refute his prior contentions and has carried on his discussion on this basis. Actually the paper's sole purpose was to present the mixer test as one easily performed and worthy of trial and to present by illustrations from actual field records the type of information which could be obtained. A discussion of the relative merits of different procedures, specifications and equipment was beyond the range of its data.

For any lack of clearness as to the paper's objective the authors apologize. About one half of the discussor's remarks relate to earlier arguments and data and to his interpretation of the mixing period which should have been used at Grand Coulee. Since the authors did not collaborate in the prior paper, and since they specifically exempted from the scope of this one a discussion of the specification of mixing time, a subject involving too many considerations of economics, job control, type of mixer, and opinion to be treated in a short paper, they feel that a reply to those portions of the discussion is inappropriate. On the other hand, few engineers are better qualified or more entitled to have an expert opinion on the specification of a mixing time than Mr. Abrams. His recommendations referring to a specific job involving a 4-yd. mixer, made in the light of a critical analysis of the test data and a personal knowledge of local conditions, should be carefully considered by anyone charged with writing specifications for similar conditions. In this connection the words of that dean of concrete technology, Arthur Talbot,[9] are also worthy of consideration, "The specification for time of mixing should not be based on optimum conditions of mixing. The added time for a margin of safety to cover contingencies frequently occurring

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[7] Engineer, Associate Engineer, and Assistant Engineer, respectively, U. S. Bureau of Reclamation, Denver, Colo.

[8] O. G. Patch, "Mixer Efficiency or Mortar-Mix Tests," *Proceedings*, Am. Concrete Inst., Vol. 35, pp. 173 to 180-47 (1939).

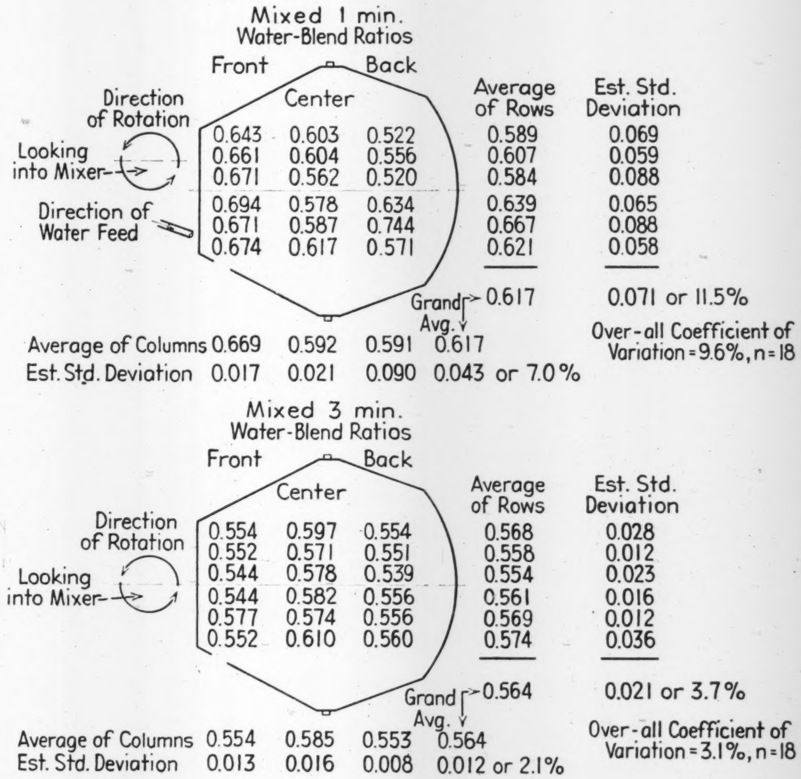
Mixed 1 min.
Water-Blend Ratios
Front
Back
Center
Direction of Rotation
Looking into Mixer
Direction of Water Feed
0.643 0.603 0.522
0.661 0.604 0.556
0.671 0.562 0.520
0.694 0.578 0.634
0.671 0.587 0.744
0.674 0.617 0.571
Average of Rows
0.589
0.607
0.584
0.639
0.667
0.621
Est. Std. Deviation
0.069
0.059
0.088
0.065
0.088
0.058
Grand Avg.
0.617
0.071 or 11.5%
Average of Columns 0.669 0.592 0.591 0.617
Est. Std. Deviation 0.017 0.021 0.090 0.043 or 7.0%
Over-all Coefficient of Variation=9.6%, n=18
Mixed 3 min.
Water-Blend Ratios
Front
Back
Center
Direction of Rotation
Looking into Mixer
0.554 0.597 0.554
0.552 0.571 0.551
0.544 0.578 0.539
0.544 0.582 0.556
0.577 0.574 0.556
0.552 0.610 0.560
Average of Rows
0.568
0.558
0.554
0.561
0.569
0.574
Est. Std. Deviation
0.028
0.012
0.023
0.016
0.012
0.036
Grand Avg.
0.564
0.021 or 3.7%
Average of Columns 0.554 0.585 0.553 0.564
Est. Std. Deviation 0.013 0.016 0.008 0.012 or 2.1%
Over-all Coefficient of Variation=3.1%, n=18

FIG. 2.—Distribution of Water-Blend Ratio in Top of Batch, Dam A. (Plotted from data for dry mix in Table III.)

NOTE.—Blend is material passing No. 100 sieve: 11 per cent rock flour, 15 per cent pumicite, 74 per cent cement. Specific gravity of blend = 3.02. Plant water-blend ratio = 0.54. Mix 1:3.0:9.0 with 20 per cent pumicite by weight of cement.

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[9] Arthur N. Talbot, "Research on Mixing of Concrete in Batch Mixers," *Proceedings*, Highway Research Board, eceDmber, 1928, p. 41.

that affect quality should be sufficiently large to insure high-grade quality under any ordinary circumstances, unless the scrutiny and supervision is to be more active and severe and farseeing than is to be expected even on the best regulated work."

The remainder of the discussion falls roughly under the following headings:

1. Failure to present original data and description of tests.
2. Sampling.
3. Statistical measures and concepts.
4. Ball mills and concrete mixers.
5. Statements with which the authors agree.

1. *Failure to Present Original Data and Description of Tests.*—Repeated criticisms dealing with lack of original data and descriptive matter can to some extent be met in this closure. Space has been granted for sample primary data and for notes on test procedure, which will be found in the Appendix to this closure. That the data were not given originally was due to the same difficulty Mr. Abrams met in 1919 in presenting his famous water-cement ratio law.[10] "In a brief report of this kind it is impracticable to present more than an outline of the methods of applying the principles to practical problems; in only a few cases are experimental data given on which these conclusions are based." It is fortunate indeed for concrete technology that his limitations as to data presented did not lead readers to the rejection of his conclusions.

2. *Sampling.*—The discussor attaches more importance to the method of sampling than do the authors. If concrete is mixed until the variation between samples is well within the limits of testing error, then any three samples should give about the same result. On the other hand if a definite range of variability greater than experimental error is acceptable, the method of sampling should be clearly stated.

The variability requirement suggested in the authors' paper, a range in unit weight of three samples not greater than 2.3 lb., for a single test contemplated the collection of samples from the discharge of the mixer in three 6-qt. buckets. The first sample from the discharge to be taken represents material from the near top of the batch; the second, material from the middle; and the third, material from the last of the discharge, and coming from the back bottom of the batch. This method of sampling interferes but little with the production of concrete and tends to represent extremes of variability. This was the method by which the data given for dam B in Fig. 1 were obtained. Each plotted point represented the average of several different batches and a different set of batches was used for each different mixing period.

A second method of sampling was utilized at dams A, C, and D where the purpose of the tests was to determine the complete batching and mixing performance of the mixer, rather than tests for control. The top of a single batch of concrete was sampled 18 times (accompanying Fig. 2) with the mixer stopped after 1 min. and again after 3 min. of mixing. Working from left to right, facing the discharge, six spot samples from each row sufficient for 2 lb. of mortar were shoveled into separate containers from the front, the center, and the back of the mixer.

Either of the two sampling methods will indicate differences in uniformity of the batch and can be used to improve or control the mixing process. Generally the average variation found can be expected to be greater than the average typical of the batch.

3. *Statistical Measures and Concepts.*—In computation and presentation of data, the authors have endeavored to follow the clear treatment given in the A.S.T.M.

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[10] Duff A. Abrams, "Design of Concrete Mixes," *Bulletin No. 1*, Lewis Institute (1919).

Manual on Presentation of Data[11] and other standard works on statistical methods. Since much of the discussion is based on different concepts and methods of computation, it is impractical to cover all points of disagreement in a short closure. A few of the more important points raised will be discussed briefly.

(*a*) Dealing with the use of suitable units in which to measure variability, the discussion states, "Mean variation has long been used and is well adapted to this purpose."

The A.S.T.M. Manual, referring to measures suitable for expressing variation of sets of data consisting of a small number of observations, states, "Because of ease of computation, the control chart for range, R, is frequently used as an economical substitute for the control chart for standard deviation, σ, more particularly in routine inspection work." Shewhart[12] shows that with sets of only three samples both range and average deviation (mean variation) are 99.5 per cent as efficient as standard deviation and "For a sample of four, practically all the information contained in the data is retained by using the range."

(*b*) Dealing with the relationship between statistical measures, "A range of 5 per cent is usually expected to give a coefficient of variation of about 1.6 per cent." From the A.S.T.M. Manual, on the contrary, the expected coefficient of variation is about 3 per cent.

(*c*) Concerning the mixing equation, the discussion states, "In Figs. 1 and 2 large experimental errors were indicated for 20, 30, 60 revolutions of the mixer drum, but no errors for 5 or 10 revolutions, although the equations of the hypothetical curves show constant errors." It was briefly explained under *Equation for Mix Variability* and under *Required Mixing Period*, that where an observed variation R_1 is due to two independent causes of variation, true variation R_2 and experimental error R_3, the observed value is $R_1 = (R_2^2 + R_3^2)^{\frac{1}{2}}$ and not the arithmetic sum of R_1 and R_2. Referring to Fig. 2, the true range of variability of the unit weight of mortar at 10 revolutions was 2.9 lb. and the experimental error range was 1.1 lb. The observed range of variation was therefore $(2.9^2 + 1.1^2)^{\frac{1}{2}} = 3.1$ lb., a value only 0.2 lb. larger than the true variation though the experimental error remained at 1.1 lb., the same as at 60 revolutions. This small difference cannot be shown on the graph.

(*d*) Also, "Statistical theory has its place . . . but there is no assurance whatever that it applies to a single sample lifted at random out of a universe"; and in another connection, "A high-range mix may indicate imperfect mixing, but such a test is ignored unless it is substantiated by a check test"; still again "The authors assumed . . . a zero range in unit weight of three samples, represents perfectly mixed concrete," and finally, "Acknowledged lack of control . . . make the reported values worthless as a basis for usable conclusions." Statements such as the foregoing indicate the authors should not have relied so much on the reader's familiarity with the A.S.T.M. Manual for explanation of their basic concepts.

A 4-yd. batch of concrete contains enough mortar to yield successively some 1500 2-lb. samples. The discussion has pointed out that potentially each sample has some billions of measurably different combinations of water, cement, and sand. It is evident that measurements on a single set of three samples, as compared to all possible ones, can only yield an *estimate* of the unknown value characteristic of its whole. However, statistical methods make it possible, for a given number of samples, to place mathematical odds on the likely magnitude of the error of the estimate. For a batch mixed under controlled conditions to a uniformity such that the standard deviation of unit weight is only 0.54 lb. (equivalent range of three 0.9 lb.), the estimate of the probable unit weight of the batch as a whole made from tests on three specimens might be, for example, $140 \pm \frac{0.54}{\sqrt{3}}$. That is, two out of three of repeated sets of such tests should give the weight of the batch within a margin of error ±0.22 per cent. However, if the property being investigated is an *index of variation;* that is, if it is desirable to know what mean variation would be found between all 1500 samples if they could all be tested, then an estimate of the average index of variation made from tests on a set of only three samples has an error of about 50 per cent (formula given in paper). Thus the estimate might be written $\bar{R}_3 = 0.9$ lb. ± 50 per cent. Somewhat loosely it could be stated that with more tests there is only one chance in three that the limits 0.45 — 1.35 would fail to include the true average. Reference to tables[13] likewise shows but one chance in ten for the true average to be found outside the limits 0.2 to 1.8 lb., and but once in a hundred, on the average, would the value be outside of the control limits 0.08 to 2.3 lb. An American War Standard[14] states "If not more than one out of 35 successive points, or not more than 2 out of 100 fall outside the 3-sigma control limits, a high degree of control may ordinarily be assumed. . . ."

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[11] "A.S.T.M. Manual on Presentation of Data," Am. Soc. Testing Mats., (1943). (Issued as separate publication.)

[12] W. A. Shewhart, "Economic Control of Quality of Manufactured Product," D. Van Nostrand Co., Inc., New York, N. Y., pp. 287, 388 (1931).

The authors, therefore, do not consider that a sample of zero range means a perfectly mixed batch, nor that a single sample of greater than 2.3-lb. range means the mixing process ought to be immediately changed, unless there are other supporting data.

(*e*) The discussor presents a trilinear diagram (Fig. 1) which illustrates in a clear manner the history of changes through which a unit volume of mortar goes during mixing as it approaches its one final weight and composition. Unfortunately the accompanying computation and discussion may leave the noncritical reader with a totally erroneous impression of the situation. There are 35 × 10^9 ways, according to the discussion, in which three samples may show uniform unit weight although their true composition is nonuniform with consequent variation in water-cement and strength properties. But no computation was given of the much larger figures representing the number of ways in which the unit weights and composition may be different and be so shown by the test. Both the number of ways in which an event can happen and in which it cannot happen are necessary if a computation of the above sort is to have a practical meaning.

(*f*) Faulty presentation of probabilities again occurs discussing correlation between unit weight and strength (water-cement ratio). No concrete engineer is likely to make the mistake of thinking that every change in unit weight means a change in strength. The low correlation coefficient between average unit weight and strength given in Fig. 3, though statistically significant, shows as a matter of fact that only about one third of the causes of variation of strength and unit weight were common to both. The utility of the unit weight test, however, does not depend on the correlation of average weight with strength. If any property of a sample taken from a batch of concrete is at first highly variable and then progressively becomes more uniform as mixing is improved or increased, then a measure of its average variability may be used to control the mixing process. Since the *variabilities* of most properties of fresh concrete *decrease* with increased mixing, it follows that the *averages of variability indices* will correlate with one another. High correlation of this type is shown in the graphs of Fig. 7 where individual points are based on averages of seven or more sets of tests. Though the discussor questions the validity of the strength ranges there shown, if Hayden's statement[15] "Test specimens made of concrete mixed 15 sec. may show a mean variation of 30 per cent in strength whereas those mixed 2 min. should vary less than 10 per cent" be converted to the units of the present paper, strength ranges of about 65 and 20 per cent are obtained at 4 and 32 revolutions, respectively, which conform with the general indications of the present tests.

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[13] E. S. Pearson, "The Application of Statistical Methods to Industrial Standardization and Quality Control," British Standards Inst. (London) (1935).

[14] A.S.A. Z.1, 1942, "Control Chart Method of Controlling Quality During Production," Am. Standards Assn. (1942).

4. *Ball Mills and Concrete Mixers.*—The discussion expresses concern about the amount of -100 sand ground in a mixer during the first 3 min. By selecting only a portion of the data from the quoted reference dealing with slump and by assuming that all loss of slump was due to production of -100 fines an average grinding rate was computed equivalent to 3.8 per cent per min. in terms of total sand. The only two series of chemical determinations of fine sand made during the present mixer tests showed grinding rates of 0.9 per cent for dam A and 2.3 per cent for dam D,[16] values but 25 and 60 per cent, respectively, of Mr. Abrams' computation. He supported the value of 3.8 per cent by stating that a batch of concrete in a mixer makes "an ideal grinding machine." Reference to the paper "Ball Mill Grinding"[17] leaves no doubt as to the similarity of the two mechanisms, in appearance, speeds, and charges, but the adjective "ideal" in this application does not apply. When a ball mill is wet-charged and run like a mixer, its output in terms of specific surface of particles produced per minute drops to about 10 to 30 per cent of its optimum. At the percentages of critical speeds usual for mixing concrete the grinding mill's batch motion is described as "a slow, sluggish, rolling action," typical of action in a concrete mixer, but not productive of the highest grinding output.

The phrase "critical speed" needs additional explanation. If two concrete mixers of similar design but different maximum diameters are gradually increased in speed, measured in rpm., they will each arrive at a centrifuging velocity at which the finest particles in contact with the drum will travel with it and no longer fall. At this critical speed, given by the formula $\frac{265}{d^{\frac{1}{2}}}$, d being the maximum diameter of the drum in inches, the mixing process in two mixers may be said to be to a degree similar since the finest particles, at least, behave alike. At intermediate percentages of critical speeds likewise some correspondence of behavior can be expected. The bulletin on ball mill grinding states "In spite of the lack of a close analogy between the deportment and the formula, critical speed is a valuable anchor in comparing mills of different diameters. Some people take critical speed too seriously." The same statement no doubt applies to concrete mixers. On the other hand the authors know of no better variable against which to plot mixer performance. There seems to be no reason to expect that "time," in itself and without reference to rpm., has any effect on the mixing. In so far as the authors' tests are competent, they show, when plotted against revolutions, the performance of different diameter mixers at different speeds tends to be somewhat similar, reducing the instantaneous variability of the batch at a rate of 14 to 28 per cent per revolution (mixing coefficient *B*, Fig. 1).

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[15] Arthur C. Hayden, "Concrete and Reinforced Concrete," Am. Civil Engrs' Pocket Book, Fifth Edition, John Wiley and Sons, Inc., New York, N. Y., p. 1029 (1930).

[16] Each value is the average of tests of seven different batches mixed 3 min.

[17] W. H. Coghill and Fred D. Devaney, "Ball Mill Grinding," *Technical Paper No. 581*, U. S. Bureau of Mines (1937).

5. *Statements With Which the Authors Agree.*—Rather than continue to comment on the large portion of the discussion with which the authors cannot agree, it is believed that the reader will derive more benefit by noting the few points on which substantial accord is obtained and by having additional pertinent tests presented.

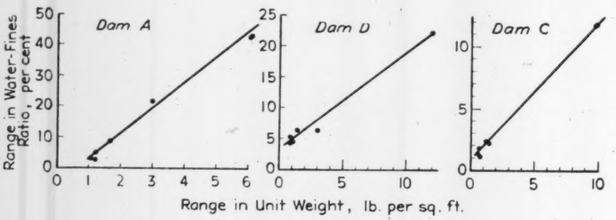

FIG. 3.—Relation of Water-Fines Ratio to Unit Weight.

(*a*) Mr. Abrams states "I have no quarrel with the *data* secured in the Patch test; they give a proper measure of mixer efficiency, but they were improperly used and interpreted by the U.S.B.R." To the first two of these three statements the authors fully assent. The test, with a slight modification,[18] is still being currently used on several Bureau projects. The basic data provided by the Patch test is shown in the accompanying Fig. 2 in terms of water-blend ratio,[19] the data coming from dam A. To make the computations, 3.2 per cent of −100 sand was deducted from the weights of material passing the No. 100 sieve at both the 1-min. and 3-min. mixing periods since the change due to grinding was unknown. The variations shown in water-blend ratio may be compared with those shown in Fig. 6 for the dry mix, the variations in this latter case being expressed in terms of unit weight. Note in the accompanying Fig. 2 the magnitudes of the water-blend ratios in relation to the point at which water enters the batch. Observe that the standard deviations for sets of tests in the plane of rotation of the mixer blades are consistently less than for sets of tests from front to back of the mixer.

Note the consistency of the ratios and the standard deviations. This type of information would be completely lost if all variations were lumped into a single index as suggested in the discussion. Finally observe the large changes in water-blend ratio between 1 and 3 min. and especially note the reduction in the values of the standard deviations. Regardless of different interpretations as to causal factors there is no doubt the test directly measures an increase in uniformity of the batch.

(*b*) Mr. Abrams has suggested that an index of variation based on the water-fines[20] ratio is an acceptable unit for measuring mixer performance. Excellent agreement of ranges in unit weight with ranges in water-fines is shown for three mixers in the accompanying Fig. 3.

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[18] Tests made in accordance with the Third Edition of the Concrete Manual, using Method A (limit of 10 per cent on water-cement ratio and sand-cement ratio) in one instance and method B (limit of 10 per cent on water-fines ratio) in other cases.

[19] Blend consists of portland cement plus 20 per cent pumicite by weight as pointed out by Mr. Abrams.

[20] "Fines" are defined as all material passing No. 100-mesh sieve.

Evidently either index discloses much the same information. On the graphs have been indicated the ranges in weights and water-fines found after 1 min. of mixing. They could be used for specification purposes if the degree of uniformity there shown is considered satisfactory, as has been suggested by Mr. Abrams.

(*c*) There is no doubt that the average magnitude of the numerical index of variation at any given mixing time depends on the sampling method. Collecting the sample from a ribbon discharge tends to produce greater uniformity than taking a spot sample. Differences in sampling quite possibly account for part of the differences in

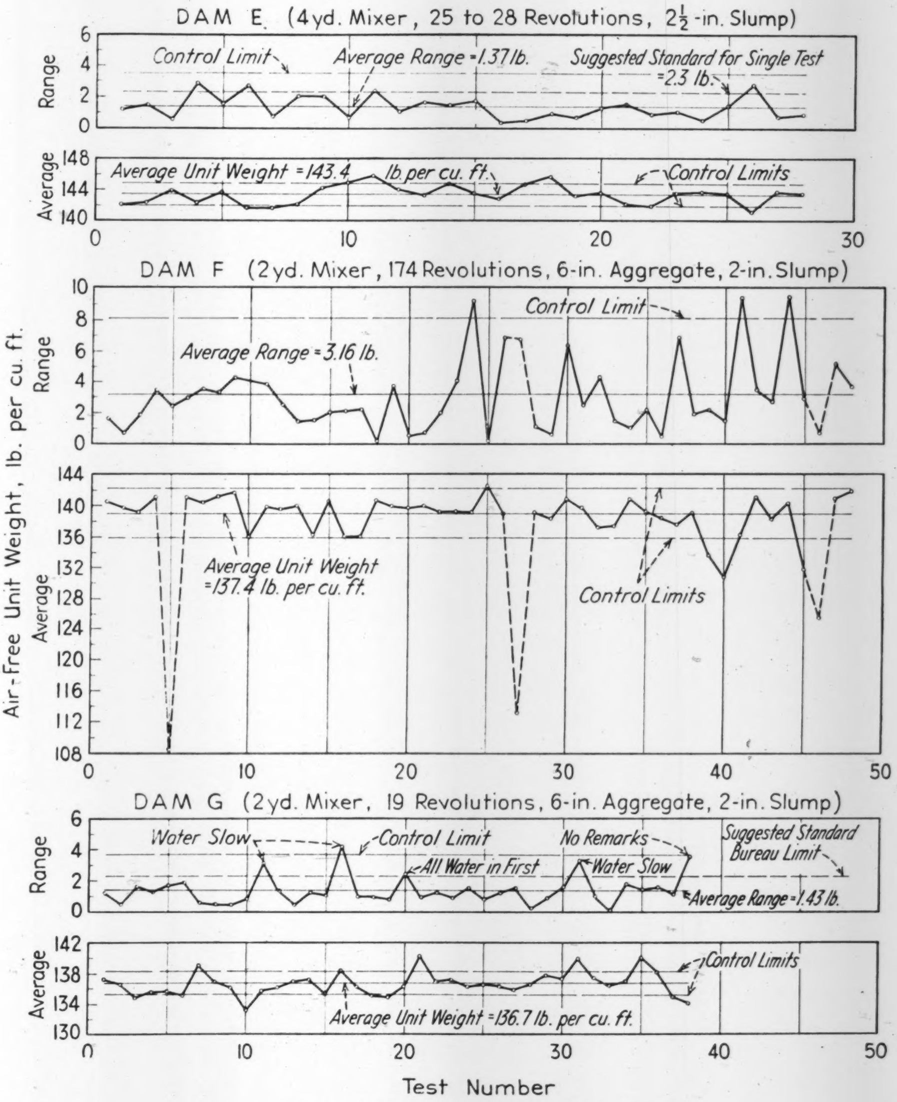

FIG. 4.—Control Charts for Air-Free Unit Weight.

uniformity shown for the different mixers of Fig. 1 of the paper.

(*d*) The authors and the discussor are agreed in noting the effect of variations in slump on results shown by the mixer tests.

(*e*) The tests show differences in the degree of uniformity produced after the same amount of mixing at dam A and dam B, and Mr. Abrams has suggested a possible explanation. Even larger differences are shown for the three mixers in the accompanying Fig. 4, the mixers at dams E and F being from projects other than the Bureau of Reclamation. Compare the mixer at dam E, mixing to a range in weight of 1.37 lb. at 28 revolutions with that at dam F whose concrete varied by 3.16 lb. after 17 revolutions of mixing and whose cylinders had a coefficient of variation of strength of 20 per cent as compared to 10 to 12 per cent typical of better controlled jobs. Or compare the latter with a similar size mixer at dam G mixing 19 revolutions but with a variability in weight as low as 1.43 lb. While this value is considerably above the average value of 0.9 lb. suggested as a standard, reasons for the variations taken from the field notes are noted on the graph.

The authors wish to close their discussion with the comment that while they have been unable to accept many of Mr. Abrams' interpretations and criticisms of the mixer test, the fact that both he and the authors have agreed that the test shows significant differences for different types of mixes and between different mixers, seems ample warrant for using it until something better is available. The authors believe that a trial will prove the test to be an inexpensive and efficient aid to concrete control; that even before the results of compression tests are known, information can be made available as to the probable uniformity of production. Finally the test may be used to guide mixer improvements. A study of ball mill literature indicates that grinding of fine sand is a function of total revolutions and not speed. If mixing also follows the same law, faster mixing should be possible by running mixers at a higher percentage of critical speed than is now current. Future experimentation will determine the answer.

APPENDIX

1. *Notes on testing.*—The accompanying Fig. 5 shows pycnometers[21] used to determine the specific gravity of a sample of minus No. 4 mortar. After obtaining the air weight of each sample, the pycnometers are filled up to the neck with water at about the same temperature as the mortar. (The volume of the pycnometers also should be known at the same temperature.) The content of each pycnometer is then agitated gently by rolling to remove entrapped air and filled to the top with water.[22] On a recent job, results shown in Fig. 4 of the authors closure, dam G, uniform removal of air from the mortar was facilitated by use of vacuum during the period of agitation. By screwing the inverted funnel down to a fixed mark against a rubber washer, a constant volume of the pycnometer is obtained. Another method of obtaining constant volume, is to grind the top of the jar smooth so that a piece of plate glass can be used as a cover. The jar is filled until the meniscus is inverted and the plate glass slid on to remove bubbles and obtain constant volume. It is particularly important to treat all samples alike, both with respect to manipulation and to time of observation. Hydration changes the observed specific gravity of the cement enough to be detected in a period of

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[21] Similar pyconometers have been described by R. W. Crum as pointed out in the "Significance of Tests of Concrete and Concrete Aggregates," Am. Soc. Testing Mats., Second Edition, p. 132 (1943). (Issued as separate publication.)

[22] Although not mentioned in published literature, mortar testers on all Bureau jobs have always stirred or agitated the mortar samples to eliminate entrapped air before obtaining the immersed weight in the Patch test.

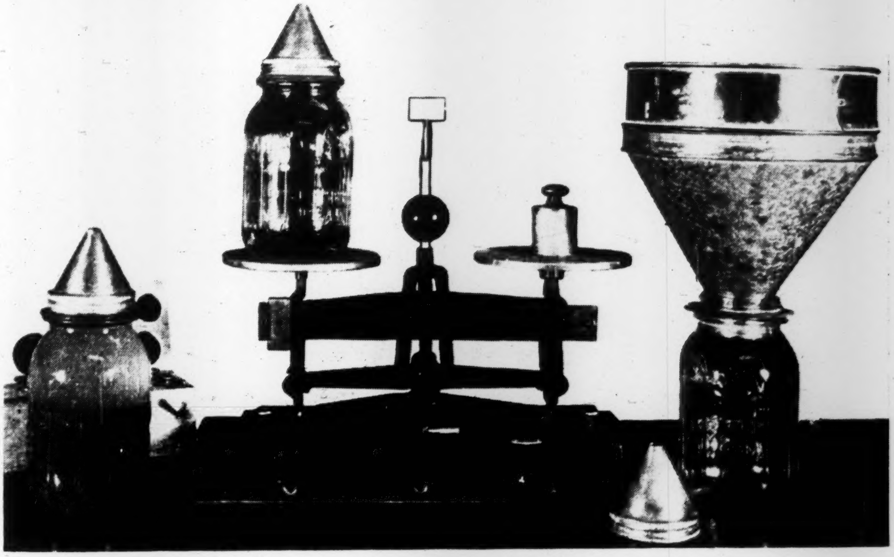

FIG. 5.—Mortar Testing Equipment.

TABLE I.—MORTAR SAMPLES OBTAINED AT MIXING PERIODS OF ½ TO 6 MIN., DAM A.

Summarized in Figs. 2 and 5 of original paper.

Specific gravity of sand 2.68, cement 3.20, and pumicite 2.35. Maximum size of aggregate 8 in. Cement content 0.80 bbl. per cu. yd.

Mix proportions 0.2:1:3.2:8.8 by weight of pumicite, cement, sand, and gravel, respectively.

Samples taken from top of batch while mixer was stopped. Total period of test 15 to 25 min.

A period of 20 to 30 min. was required to obtain samples from each batch.

	Item[a]	Mixing Time—½ min.			Mixing Time—1 min.			Mixing Time—1½ min.			Mixing Time—2 min.			Mixing Time—3 min.			Mixing Time—6 min.		
		Location in Mixer			Location in Mixer			Location in Mixer			Location in Mixer			Location in Mixer			Location in Mixer		
		Front	Center	Back	Front	Center	Back	Front	Center	Back	Front	Center	Back	Front	Center	Back	Front	Center	Back
Date: 3-25-41	A	574	860	814	653	582	628	910	774	754	723	788	768	742	690	779	710	749	775
Batch A-1	B	322	484	455	361	324	352	502	431	422	402	438	426	413	384	434	395	417	433
Plant w/c 0.68	C	235	348	305	258	232	243	353	302	294	277	305	296	286	264	299	270	285	297
Date: 3-26-41	A	566	524	584	506	528	567	612	625	630	606	662	530	578	563	666	608	511	724
Batch A-2	B	326	301	332	290	302	321	348	355	355	340	373	304	325	316	370	344	288	410
Plant w/c 0.68	C	223	228	260	207	226	240	253	257	251	237	262	216	228	220	263	239	198	284
Date: 3-31-41	A	556	501	430	634	552	576	688	671	512	752	682	801	694	746	806	788	795	894
Batch A-3	B	321	283	251	353	309	326	382	374	289	416	376	446	389	417	452	440	445	498
Plant w/c 0.69	C	249	192	179	252	217	230	266	259	203	290	265	316	265	285	310	296	300	338
Date: 4-2-41	A	684	390	511	771	725	666	780	849	829	747	784	698	785	743	723	817	761	788
Batch A-4	B	389	218	282	428	406	370	437	477	465	415	436	389	437	413	405	456	427	444
Plant w/c 0.65	C	310	149	180	311	284	257	300	337	326	283	303	266	302	288	284	309	291	301
Date: 4-3-41	A	747	528	389	817	841	764	1007	968	881	701	679	742	775	998	868	925	847	995
Batch A-5	B	425	299	223	456	471	429	563	543	496	392	380	417	431	559	485	519	478	562
Plant w/c 0.68	C	338	211	142	328	341	310	390	383	349	269	263	293	300	390	339	352	328	385
Date: 4-7-41	A	562	735	710	730	719	636	739	870	783	711	860	666	771	847	771	724	757	800
Batch A-6	B	324	362	398	405	406	356	413	489	440	398	481	375	434	475	434	404	423	447
Plant w/c 0.66	C	279	280	282	304	310	234	296	356	312	285	336	267	305	335	305	276	290	306
Date: 4-9-41	A	671	543	587	738	658	608	657	745	711	656	771	633	800	649	796	713	477	708
Batch A-7	B	382	310	336	408	370	341	367	420	399	367	432	353	448	364	446	396	265	393
Plant w/c 0.69	C	325	260	289	313	250	229	265	297	267	258	302	246	310	252	309	266	180	266

[a] Item A = Air weight of mortar sample, grams.
Item B = Immersed weight of mortar sample, grams.
Item C = Immersed weight of sand passing the No. 14 sieve and retained on the No. 100 sieve, grams.

Table II.—MORTAR SAMPLES AND CONCRETE STRENGTHS OBTAINED AT DAM B AFTER 1½ AND 2½ MIN. OF MIXING

Summarized in Figs. 3 and 4

Specific gravity of sand 2.67, cement 3.04. Maximum size of aggregate 6 in.
Mix proportions 1:2.5:7.2. Plant w/c minus 0.070 = net w/c.

		Mixing time 1½ min. (15 revolutions)							Mixing time 2½ min. (25 revolutions)				
	Item[a]	Sampled from Discharge			28-day Strength, Single Cylinders, psi.			Item[a]	Sampled from Discharge			28-day Strength, Single Cylinders, psi.	
		First	Middle	Last					First	Middle	Last		
Date: 10-16-39-S	A	944.0	1156.0	1209.0			Date: 11-5-39-N	A	1080.0	996.0	1107.5		
Plant w/c 0.613	B	532.5	651.0	682.0	4175	3850	Plant w/c 0.618	B	613.0	566.5	628.0	4385	4360
Slump 1.75 in.	C	354.0	432.5	463.0			Slump 1.75 in.	C	404.0	371.5	407.0		
Date: 10-17-39-N	A	1150.0	1317.0	1110.0			Date: 11-5-39-D	A	993.0	937.0	1381.0		
Plant w/c 0.635	B	644.0	738.5	623.5	4175	4145	Plant w/c 0.589	B	562.5	532.5	781.5	3680	3935
Slump 2.75 in.	C	423.5	485.0	406.0			Slump 2.25 in.	C	371.0	353.0	509.0		
Date: 10-17-39-S	A	957.5	976.0	660.0			Date: 11-5-39-D	A	1137.0	1141.5	1119.5		
Plant w/c 0.627	B	540.0	550.0	374.5	4050[b]	4340[b]	Plant w/c 0.611	B	645.0	648.0	636.0	3945	3980
Slump 1.5 in.	C	359.0	369.5	255.0			Slump 2 in.	C	423.0	427.0	419.0		
Date: 10-18-39-N	A	1108.5	1137.5	1151.0			Date: 11-5-39-S	A	1190.5	1178.5	1210.5		
Plant w/c 0.650	B	625.0	643.0	655.0	4440	4140	Plant w/c 0.635	B	668.0	666.5	684.0	3855	3840
Slump 2.25 in.	C	405.0	424.0	440.0			Slump 2 in.	C	438.5	433.0	446.5		
Date: 10-18-39-D	A	948.0	1127.0	1258.0			Date: 11-6-39-N	A	847.5	865.0	815.0		
Plant w/c 0.626	B	535.0	639.0	713.0	3705	3785	Plant w/c 0.593	B	480.0	490.0	461.0	4120	4100
Slump 2 in.	C	357.0	428.0	481.0			Slump 2.25 in.	C	319.5	323.0	299.5		
Date: 10-18-39-D	A	825.0	1102.2	996.8			Date: 11-6-39-D	A	1015.0	1489.0	1100.5		
Plant w/c 0.640	B	473.3	633.3	571.1	4670	4685	Plant w/c 0.614	B	573.0	838.5	618.5	4040	4075
Slump 1 in.	C	315.0	424.8	390.3			Slump 2.25 in.	C	371.5	541.0	405.5		
Date: 10-18-39-S	A	1067.0	974.0	1002.0			Date: 11-6-39-S	A	1167.0	1155.0	1343.5		
Plant w/c 0.638	B	599.0	544.0	560.0	3900	3640	Plant w/c 0.632	B	657.0	650.0	754.5	3465	3360
Slump 2 in.	C	394.0	362.0	377.0			Slump 1.5 in.	C	431.5	426.5	496.0		
Date: 10-19-39-N	A	1266.5	1275.0	1276.5			Date: 11-7-39-N	A	843.7	787.6	1159.4		
Plant w/c 0.622	B	711.0	713.5	720.0	3110	2995	Plant w/c 0.62	B	479.1	446.9	657.7	4000	3560
Slump 3 in.	C	471.5	476.5	491.0			Slump 2.5 in.	C	317.6	293.2	433.9		
Date: 10-19-39-D	A	972.0	978.5	1013.0			Date: 11-7-39-D	A	1078.5	960.0	974.0		
Plant w/c 0.643	B	549.0	553.0	571.5	3620	3635	Plant w/c 0.627	B	610.0	543.0	551.0	4340	4120
Slump 2 in.	C	368.5	374.5	392.5			Slump 2.25 in.	C	382.0	347.5	359.0		
Date: 10-19-39-S	A	1008.0	1018.0	1030.0			Date: 11-7-39-S	A	1221.0	1169.5	1027.5		
Plant w/c 0.615	B	563.0	568.0	573.0	3600	3650	Plant w/c 0.618	B	689.5	660.5	581.5	3890	3710
Slump 2 in.	C	370.0	382.0	379.0			Slump 1.5 in.	C	446.5	426.0	382.5		
Date: 10-20-39-N	A	1193.0	1137.0	1083.0			Date: 11-8-39-N	A	685.5	910.0	974.0		
Plant w/c 0.641	B	670.5	639.0	610.0	3555	3485	Plant w/c 0.630	B	388.5	515.5	550.5	3870	3865
Slump 2.25 in.	C	451.5	425.5	406.0			Slump 1.75 in.	C	252.5	333.0	355.5		
Date: 10-20-39-D	A	1214.5	1170.5	954.0			Date: 11-8-39-D	A	1113.5	1021.0	1070.0		
Plant w/c 0.651	B	684.5	663.0	541.0	3920	4070	Plant w/c 0.650	B	629.0	574.5	604.0	3720	3740
Slump 1.75 in.	C	452.0	444.5	367.0			Slump 1.75 in.	C	406.0	374.0	396.5		
Date: 10-20-39-S	A	981.0	1012.0	1134.5			Date: 11-8-39-S	A	1116.5	1247.0	1165.0		
Plant w/c 0.613	B	554.0	572.0	639.5	4075	3945	Plant w/c 0.622	B	626.0	702.0	657.5	3150	3185
Slump 1.75 in.	C	370.0	378.0	431.0			Slump 2.5 in.	C	407.0	457.0	431.0		
Date: 10-21-39-N	A	775.5	565.5	955.5			Date: 11-9-39-N	A	968.0	921.5	958.5		
Plant w/c 0.622	B	433.0	316.5	535.0	3370	3475	Plant w/c 0.631	B	547.5	520.5	543.5	3545	3370
Slump 2.75 in.	C	283.0	207.0	352.0			Slump 2.5 in.	C	359.0	339.0	357.5		
Date: 10-21-39-D	A	914.5	945.5	931.5			Date: 11-9-39-D	A	1029.0	990.0	775.5		
Plant w/c 0.662	B	512.0	530.0	522.5	3870	3990	Plant w/c 0.602	B	582.0	559.5	439.0	4650	4475
Slump 2.75 in.	C	325.0	344.5	348.5			Slump 2.25 in.	C	381.5	363.0	291.0		
Date: 10-21-39-S	A	1174.5	1129.5	960.0			Date: 11-9-39-D	A	983.0	1144.0	1134.0		
Plant w/c 0.620	B	660.0	633.5	538.0	2925	3040	Plant w/c —	B	557.0	647.0	642.0	4490	4330
Slump 2.25 in.	C	426.5	414.0	351.0			Slump 2 in.	C	365.0	422.0	424.0		
Date: 10-22-39-N	A	908.0	1162.5	1190.0			Date: 11-9-39-S	A	1153.0	1117.0	1108.0		
Plant w/c 0.641	B	505.0	649.5	666.0	3690	3620	Plant w/c 0.601	B	657.0	636.0	628.5	3730	3720
Slump 3 in.	C	322.5	418.5	428.5			Slump 2 in.	C	426.0	414.0	398.0		

TABLE II.—Continued.

	Item[a]	Mixing time 1½ min. (15 revolutions)						Item[a]	Mixing time 2½ min. (25 revolutions)				
		Sampled from Discharge			28-day Strength, Single Cylinders, psi.				Sampled from Discharge			28-day Strength, Single Cylinders, psi.	
		First	Middle	Last					First	Middle	Last		
Date: 10-22-39-D	A	1179.5	1202.5	1273.0			Date: 11-10-39-N	A	788.5	835.0	685.0		
Plant w/c 0.636	B	667.0	679.0	718.0	3670	4235	Plant w/c 0.620	B	449.5	478.0	392.5	4330	4120
Slump 2.25 in.	C	437.5	449.0	475.0			Slump 1.75 in.	C	296.5	316.5	263.5		
Date: 10-22-39-S	A	1193.5	1100.5	1081.5			Date: 11-10-39-D	A	1165.5	1175.0	851.0		
Plant w/c 0.622	B	667.5	619.0	609.0	3590	3635	Plant w/c 0.623	B	660.0	665.5	484.0	3765	3910
Slump 3.25 in.	C	439.0	407.0	399.5			Slump 2 in.	C	440.0	436.0	327.0		
Date: 10-23-39-N	A	1096.0	1199.0	1175.5			Date: 11-10-39-D	A	918.0	662.5	951.0		
Plant w/c 0.658	B	615.5	674.0	662.0	3630	3870	Plant w/c 0.603	B	519.0	374.0	538.0	3765	3855
Slump 2.25 in.	C	393.0	432.0	425.0			Slump 2 in.	C	347.0	249.0	361.0		
Date: 10-23-39-D	A	958.0	999.0	955.5			Date: 11-10-39-S	A	928.5	1111.0	1072.5		
Plant w/c 0.626	B	532.0	557.5	535.5	3335	3430	Plant w/c 0.601	B	529.5	633.0	612.0	3810	3855
Slump 3.75 in.	C	348.5	363.0	348.5			Slump 2 in.	C	354.5	420.0	411.5		
Date: 10-25-39-N	A	1200.0	1059.5	1032.5			Date: 11-11-39-N	A	1048.0	1097.5	1067.0		
Plant w/c 0.609	B	673.0	595.5	580.5	3775	3360	Plant w/c 0.615	B	597.0	626.5	607.5	4300	4280
Slump 2.5 in.	C	448.5	390.0	380.0			Slump 2 in.	C	396.0	417.5	392.0		
Date: 10-25-39-D	A	1161.0	882.0	1060.0									
Plant w/c 0.630	B	654.0	498.0	598.0	3740	3900							
Slump 1.75 in.	C	429.0	325.0	395.0									
Date: 10-25-39-S	A	1180.0	1048.5	1008.0									
Plant w/c 0.63±	B	664.0	590.0	565.5	3440	3415							
Slump 2.25 in.	C	433.0	388.5	374.5									

[a] Item A = Air weight of mortar sample, grams.
Item B = Immersed weight of mortar sample, grams.
Item C = Immersed weight of sand passing the No. 14 sieve and retained on the No. 100 sieve, grams.

[b] 5050 and 5340 psi. in field data; assumed to be typographical error.

15 min. Precision of the operator's technique should be known or determined before making a mixer test.

The accompanying Table I shows original gram weights recorded by the mortar tester, all weighing containers being counterbalanced. Each batch was tested six times after mixing for the intervals shown, by stopping the mixer and obtaining samples at the front, center, and back of the top of the batch. Weights A and B were used for unit weight calculations, weight C not being needed unless water-fines or water-cement and sand-cement ratios are desired. Air-free unit weight computed from the data is summarized in Figs. 2 and 5 of the original paper.

The weights shown in the accompanying Table II are from original field records. The samples were taken while the mixer was discharging. This also applies to samples for strength specimens; two 14-qt. buckets, suspended by a cable, were swung into the discharge, usually between taking the first and second sample for the mortar test. The concrete for strength specimens was wet-screened to 1½-in. maximum size, slumped, and cast into 6- by 12-in molds, using a small vibrator for compaction. Figures 3 and 4 of the original paper are based on data shown in Table II.

The accompanying Table III shows the original data which is summarized in Fig. 6 of the original paper. Concrete for slump test was taken from the top center of the batch after 3 min. of mixing. Proportions for the regular mix are given by the notes in Table I. The other mixes were obtained by altering the cement and/or water content of the regular mix.

TABLE III.—MULTIPLE SAMPLES OF VARIOUS MIXES AFTER MIXING 1 AND 3 MIN., DAM A.
Summarized in Fig. 6

Data on regular mix is given by notes in Table I. Other mixes were obtained by altering the cement and/or water content of the regular mix.

	Item[a]	Front of Mixer (left to right)						Center of Mixer (left to right)						Back of Mixer (left to right)					
		1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
DRY MIX																			
Mixing Time—1 min. (10 revolutions)																			
Date: 3-31-41 Plant w/c 0.65 Slump 0.75 in. Bbl. cement per cu. yd. 0.80	A	893	927	886	689	825	725	375	521	517	526	563	487	557	506	554	460	472	455
	B	490	511	485	376	453	398	211	292	290	297	318	274	317	287	314	255	258	257
	B	341	364	341	266	320	283	154	210	204	216	233	200	228	208	221	181	188	186
	Mixing Time—3 min. (30 revolutions)																		
	A	616	624	591	561	630	619	593	779	527	674	638	561	579	573	581	617	644	700
	B	343	348	329	313	349	345	329	431	293	373	354	310	323	320	325	344	358	390
	C	235	238	223	214	238	236	229	293	201	256	242	215	221	220	223	237	244	268
LEAN MIX																			
Mixing Time—1 min. (10 revolutions)																			
Date: 4-7-41 Plant w/c 1.02 Slump 1.25 in. Bbl. cement per cu. yd. 0.51	A	861	902	919	849	948	869	818	882	677	800	659	723	612	664	629	767	647	707
	B	474	494	502	465	518	473	454	490	377	451	367	402	344	372	354	429	363	397
	C	377	388	393	369	409	372	352	377	288	353	288	315	264	282	272	327	277	305
	Mixing Time—3 min. (30 revolutions)																		
	A	726	795	868	808	900	761	784	833	793	928	881	772	762	885	876	755	796	864
	B	400	440	480	447	496	419	430	457	434	510	483	424	421	488	482	419	441	479
	C	303	334	360	339	374	317	327	344	327	385	364	320	318	366	361	319	332	361
REGULAR MIX																			
Mixing Time—1 min. (10 revolutions)																			
Date: 3-25-41 Plant w/c 0.67 Slump 1.75 in. Bbl. cement per cu. yd. 0.79	A	832	812	761	760	604	774	595	589	548	743	746	652	510	483	650	524	566	490
	B	462	451	421	420	334	428	335	329	309	418	414	365	288	272	365	292	316	274
	C	329	322	293	291	234	300	244	236	220	308	294	259	207	199	256	208	226	199
	Mixing Time—3 min. (30 revolutions)																		
	A	672	735	564	729	631	647	742	632	695	704	665	632	640	709	777	614	578	596
	B	376	411	315	407	352	360	412	354	386	395	371	353	357	393	433	342	322	332
	C	261	283	216	276	240	248	283	246	264	272	258	246	246	274	300	235	223	230
RICH MIX																			
Mixing Time—1 min. (10 revolutions)																			
Date: 4-8-41 Plant w/c 0.44 Slump 2 in. Bbl. cement per cu. yd. 1.38	A	986	836	828	814	806	1063	627	730	791	780	636	1031	747	804	843	728	897	747
	B	551	466	462	455	450	593	348	406	440	436	353	574	411	447	470	408	503	417
	C	345	279	278	268	273	353	195	207	250	237	187	323	196	214	233	187	247	200
	Mixing Time—3 min. (30 revolutions)																		
	A	826	751	760	781	654	815	738	778	794	832	778	902	973	1000	969	812	828	971
	B	462	420	424	436	364	456	410	433	443	463	433	504	540	561	538	452	458	536
	C	258	236	238	243	203	256	230	242	245	264	241	280	301	316	302	250	257	298
WET MIX																			
Mixing Time—1 min. (10 revolutions)																			
Date: 4-3-41 Plant w/c 0.77 Slump 5 in. Bbl. cement per cu. yd. 0.78	A	948	827	762	822	843	912	1029	1019	861	725	902	854	1049	1004	949	869	981	844
	B	522	457	420	452	466	502	556	563	478	401	498	477	576	548	519	479	538	467
	C	382	327	300	323	325	350	391	392	328	284	344	335	376	362	331	306	352	308
	Mixing Time—3 min. (30 revolutions)																		
	A	731	813	873	680	859	798	897	923	878	899	907	647	988	894	694	832	948	904
	B	405	448	484	376	474	441	496	509	486	498	503	352	544	494	383	457	525	499
	C	281	307	334	250	323	302	341	351	336	346	347	247	372	334	263	313	357	342

[a] Item A = Air weight of mortar sample, grams.
Item B = Immersed weight of mortar sample, grams.
Item C = Immersed weight of sand passing the No. 14 sieve and retained on the No. 100 sieve, grams.

EFFECT OF CURING METHODS UPON THE DURABILITY OF CONCRETE AS MEASURED BY CHANGES IN THE DYNAMIC MODULUS OF ELASTICITY

By Bartlett G. Long[1] and H. J. Kurtz[1]

Synopsis

The effects of membrane curing, as well as "standard" moist curing, and no curing, upon the durability of concrete subjected to laboratory freezing and thawing, as measured by changes in the modulus of elasticity, *E*, determined by dynamic methods, are briefly described. For the purpose of comparing laboratory results with those obtained under actual field conditions, visual examinations and dynamic modulus of elasticity tests were conducted on a 3½-yr. old test wall in which the 5-ft. square concrete panels were cured with some of the membranes used in the laboratory investigation.

Under the specific conditions investigated it was found that (1) the effect of different initial curing methods upon the quality of 3½-yr. old concrete is slight, (2) the amount of surface cracking does not appear to be directly related to the method of initial curing, (3) the moisture content of laboratory specimens subjected to freezing-and-thawing conditions their behavior to a marked degree, (4) a period of drying following moist curing significantly increases the resistance of laboratory specimens subjected to freezing and thawing, (5) when the dynamic modulus of elasticity is employed as a determinant, the relative effectiveness of curing compounds may be more clearly differentiated in freezing- and-thawing tests than by tests during a curing period only, and (6) laboratory tests of concrete specimens at early ages may be difficult of correlation with the actual field behavior of concrete of greater age.

During the present war emergency the use of concrete curing compounds has developed very rapidly. These, for the most part, are resinous or waxy compounds forming thin membranes or films of varying degrees of hardness, and are used primarily to retain the mixing water in concrete. They have been used extensively in the construction work of the Corps of Engineers, U. S. Army, in curing runways for airfields and in the construction of ordnance depots, camps, hospitals, and other works. The critical materials situation, and consequent shortage of rubber hose and metal pipe, together with the fact that compound curing is usually cheaper than water curing, have contributed to the increased popularity of this method, while freedom of subgrades and excavations from excessive curing water is another important factor. Due to the relatively short time since the use of such compounds became widespread, it is to be expected that there exists very little published information concerning testing procedures and results, either from the laboratory or from observation of actual field structures. Therefore, it is hoped that the

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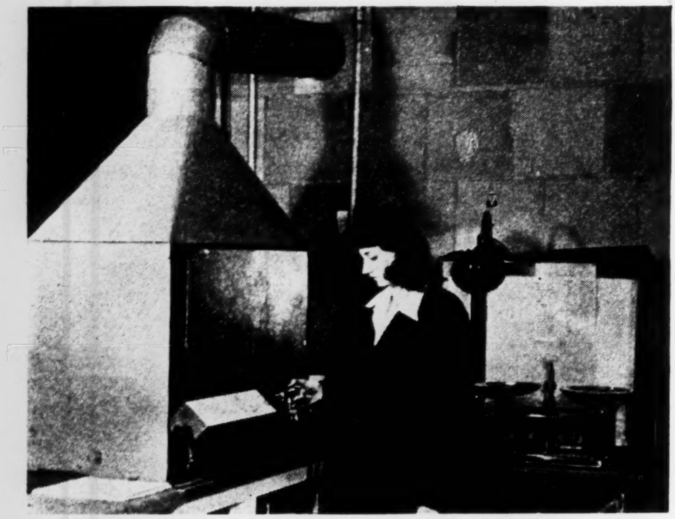
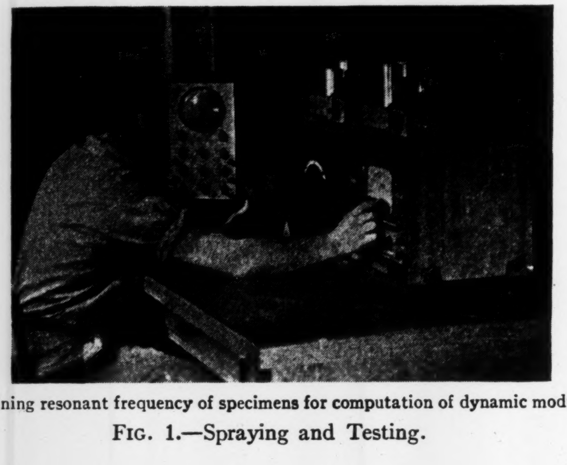


(*a*) Method of applying curing membranes to surfaces of freely supported beam specimens.

(*b*) Method of freely supporting specimens throughout curing period in controlled atmosphere of 75 F. and 50 per cent relative humidity.



(*c*) Method of determining resonant frequency of specimens for computation of dynamic modulus of elasticity.

FIG. 1.—Spraying and Testing.

data presented herein, although not sufficiently extensive to warrant other than tentative conclusions, may prove helpful to those faced with the problem of selection in concrete curing methods.

MATERIALS

Laboratory Specimens:

The concrete mix proportions used for the laboratory beam specimens (Fig. 1) were 1:3.06:4.23 by weight. Moderate heat cement (Federal Specification SS-C-206a), Ohio River sand, and Ohio River gravel were used. The aggregates have a good service record in northern Kentucky and southern Ohio. The water-cement ratio was 0.53 by weight, and the cement factor was 5 sacks per cu. yd. Sieve analyses of the sand and gravel are given in Table I.

TABLE I.—SIEVE ANALYSES OF SAND AND GRAVEL.

U.S. Standard Sieve Size	Laboratory Specimen		Test Wall	
	Per Cent Retained by Weight	Cumulative Per Cent Retained	Per Cent Retained by Weight	Cumulative Per Cent Retained
⅜ in.	...	...	0	0
No. 4	0	0	0.8	0.8
No. 8	15.0	15.0	14.4	15.2
No. 16	21.5	36.5	25.2	40.4
No. 30	27.5	64.0	28.1	68.5
No. 50	26.5	90.5	22.6	91.1
No. 100	7.5	98.0	7.8	98.9
Pan	2.0	100.0	1.1	100.0
	FINENESS MODULUS = 3.04		FINENESS MODULUS =3.15	
1¼ in.	0	0	0	0
1 in.	22	22	6.4	6.4
¾ in.	45	67	...	...
⅜ in.	33	100	83.6	90.0
No. 4	0	...	9.8	99.8
Pan	...	...	0.2	100.0

The bulk specific gravity of the gravel was 2.69; the 24-hr. and 72-hr. absorptions were 1.6 and 1.8 per cent, respectively; and the abrasion loss (A.S.T.M. Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 39)[2]) at 500 revolutions was 28.7 per cent. The gravel was submerged in water for 72 hr. previous to use, then surface dried; and the sand was laboratory air dried.

TABLE II.—CHEMICAL AND PHYSICAL PROPERTIES OF CEMENT.

Silicon dioxide (SiO_2), per cent	21.1
Aluminum oxide (Al_2O_3), per cent	5.2
Ferric oxide (Fe_2O_3), per cent	4.4
Calcium oxide (CaO), per cent	62.2
Magnesium oxide (MgO), per cent	3.7
Sulfur trioxide (SO_3), per cent	1.6
Sodium oxide (Na_2O), per cent	0.25
Potassium oxide (K_2O), per cent	0.42
Phosphorous pentoxide (P_2O_5), per cent	0.14
Manganic oxide (Mn_2O_3), per cent	0.05
Chloroform-soluble organic substances, per cent	0.004
Insoluble residue, per cent	0.10
Potential Compounds (computed):	
Tricalcium silicate ($3CaO \cdot SiO_2$), per cent	47.0
Dicalcium silicate ($2CaO \cdot SiO_2$), per cent	25.0
Tricalcium aluminate ($3CaO \cdot Al_2O_3$), per cent	6.0
Tetracalcium alumino-ferrite ($4CaO \cdot Al_2O_3 \cdot Fe_2O_3$), per cent	13.0
Calcium sulfate ($CaSO_4$), per cent	3.0
Specific surface (Wagner), sq. cm. per g.	1860
Autoclave expansion, per cent	0.10
Time of set (Gilmore):	
Initial	5 hr. 45 min.
Final	8 hr. 20 min.
Tensile strength (briquets), psi.:	
3 days	335
7 days	360
28 days	435
Compressive strength (2-in. cubes) psi.:	
3 days	1290
7 days	2020
28 days	3980

TABLE III.—PROPERTIES OF CURING COMPOUNDS—LABORATORY SPECIMENS.

Compound	Type of Solids	Solids, per cent[a]	Specific Gravity at 25/25 C.	Saponification Number, Solids	Viscosity at 25 C., centipoises	Refractive Index at 25 C.
A	Wax	29.5	0.824	97	4.6	1.451
B	Wax	28.2	0.828	89	11.0	1.453
C	Wax	44.6	0.840	94	10.0	1.458
D	Resin	40.6	0.894	11	7.2	1.499
E	Resin	49.4	0.904	157	16.5	1.485
F	Coal Tar	66.0	1.120	...	1780	...

[a] The solids were determined by heating at 105 C. for 72 hr.

Lehigh (Fogelsville) cement of the chemical analysis and properties given in Table II was used.

Certain properties of the six curing compounds used in the laboratory tests are presented in Table III.

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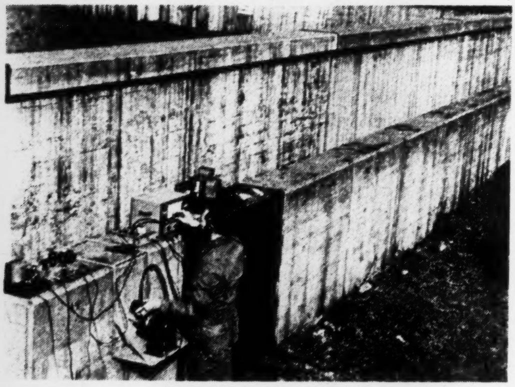
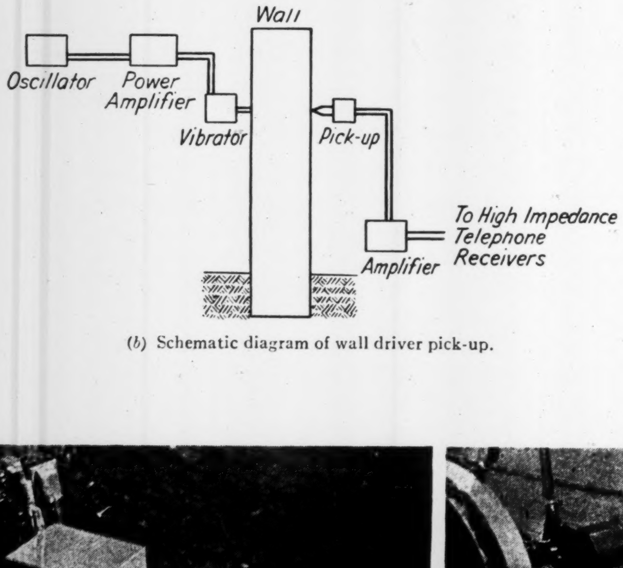
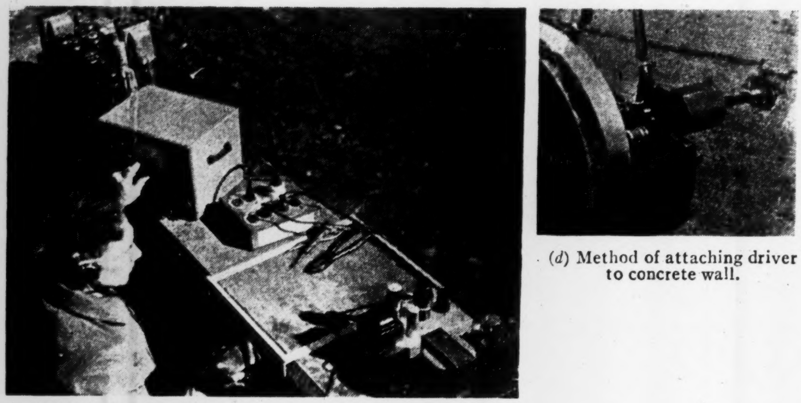
[2] 1942 Book of A.S.T.M. Standards, Part II, p. 359.



(*a*) View of test wall showing velocity determining apparatus in place.


Wall
Oscillator
Power Amplifier
Vibrator
Pick-up
Amplifier
To High Impedance Telephone Receivers

(*b*) Schematic diagram of wall driver pick-up.



(*c*) Method of determining apparent velocity of sound through concrete, for computation of dynamic modulus of elasticity.

(*d*) Method of attaching driver to concrete wall.

FIG. 2.—Test Wall.

Test Wall:

The test wall shown in Fig. 2, constructed 3½ yr. ago, is 6 ft. high, 85 ft. long, and 1½ ft. thick, the bottom extending 1 ft. below the surface of the ground. During construction, "bulkheads" of galvanized iron were placed vertically in the concrete at intervals of 5 ft., dividing the wall into 17 separate panels. The concrete mix proportions were 1:2.64:4.40 (cement: Ohio River sand: Ohio River gravel) by weight. The water-cement ratio was 0.55 by weight; and the cement factor was 5 sacks per cu. yd. Sieve analyses of the sand and gravel are given in Table I.

The cement conformed with Federal Specification SS-C-206a for cement; Portland, Moderate-heat-of-hardening. Complete analysis of the cement is not available.

At the time the test wall was constructed, the various compounds used were not carefully analyzed in the laboratory, and the chief similarity between the compounds used at that time and those of the same manufacture or name used in the present laboratory series may be in the type of solids. Compound *G*, used in curing one of the panels, is no longer manufactured and is not represented in the laboratory tests.

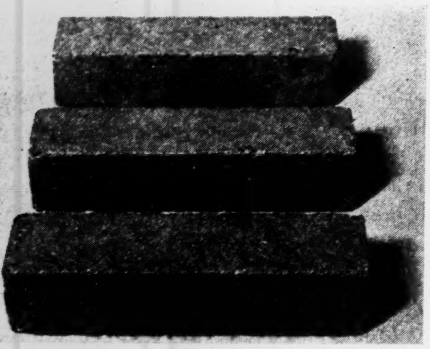
Test Methods

Laboratory Tests:

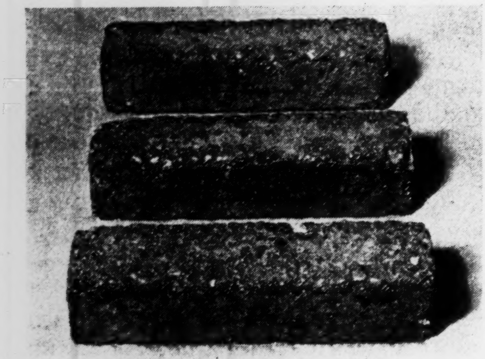
Mixing of the concrete was accomplished in a Lancaster SKG mixer and all specimens (4 by 4 by 16-in. beams) were cast in machined metal molds, in a controlled atmosphere of 70 F. and 60 per cent relative humidity. Nine groups of three specimens each were made, representing four different conditions of curing, as follows, (*a*) 14 days fog curing and 3 days air drying, (*b*) 17 days fog curing, (*c*) 18 hr. fog curing (in molds) and 16 days compound curing, and (*d*) 18 hr. fog curing (in molds) and 16 days air drying. After approximately 18 hr. in the fog room, specimens intended for "compound curing" were removed from the molds, and the compounds were applied with a DeVilbiss "air brush," at a coverage of 200 sq. ft. per gal. (Fig. 1). Calculations for coverage took into account variations in the specific gravities of the different compounds. Small steel inserts were cast into the ends of the specimens to provide free support during the compound coating operation and throughout subsequent curing.

All specimens, except the fog-cured beams, were cured in a controlled atmosphere of 75 F. and 50 per cent relative humidity, for 16 days. The fog-cured and air-dried specimens remained in the fog room for 14 days, after which they were placed with the other specimens in the same controlled atmosphere, for the three remaining days of the curing period. An additional group of three beams was fog cured for the entire 17-day period and tested immediately, in order to obtain additional information concerning the effect of variations in the moisture content of concrete subjected to freezing and thawing at early ages. Curing compounds were not removed before the specimens were subjected to alternate freezing and thawing.

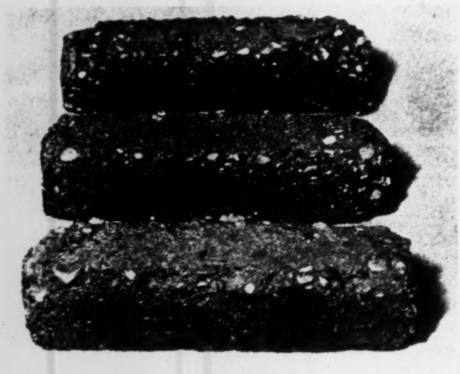
The freezing-and-thawing cycle consisted of freezing in air at −50 F. for 4 hr., and thawing in water at 40 F. for 2 hr. Previous freezing-and-thawing tests, of 4 by 4 by 16-in. beams with embedded thermocouples, have shown that the temperature of the interior of such beams reaches −35 F. in 4 hr. of freezing, and is then raised to 40 F. in 2 hr. of thawing in water at that temperature. Four cycles per day for 6 days were completed each week, the specimens remaining in the freezing room over Sundays. Tests were discontinued when the dy-



(*a*) Fog-cured and air-dried specimens after 57 cycles of freezing and thawing.



(*b*) Membrane-cured specimens (B-membrane) after 66 cycles of freezing and thawing.



(*c*) No-cured specimens after 57 cycles of freezing and thawing.

FIG. 3.—Laboratory Specimens After Freezing and Thawing.

namic modulus of elasticity had been reduced to approximately 50 per cent of the original 17-day value. Figure 3 shows the appearance of some of the beams after freezing and thawing.

Dynamic modulus of elasticity (Fig. 1) and weight change determinations were obtained throughout the curing period and subsequent freezing and thawing. The dynamic modulus was determined by the electrical method of flexural vibration employed by Hornibrook,[3] Obert,[4] and other investigators, in which a specimen is vibrated through a frequency range by means of a variable-frequency oscillator until the fundamental resonant frequency is determined. This is indicated by the maximum output voltage of a pick-up held against the specimen, and is detected by means of the corresponding maximum amplitude of the Lissajous figure displayed on the screen of a cathode-ray oscillograph.

Test Wall:

The vertical surfaces of the test wall were visually examined, and the amounts of compound remaining on the surfaces and the surface cracking of the concrete were recorded.

For the purpose of comparison of the concrete in the wall with that of the laboratory specimens, dynamic modulus determinations were made for each panel, using the longitudinal method of vibration (Fig. 2) described by Obert and Duvall.[4] The panels were vibrated on a vertical surface at a point centrally located and approximately 1.35 ft. from the top. The vibrator, a rebuilt auditorium loud-speaker, was driven by a powerful amplifier (four 6L6 tubes in push-pull parallel), and was rigidly

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[3] Floyd B. Hornibrook, "Application of Sonic Method to Freezing and Thawing Studies of Concrete," ASTM BULLETIN, No. 101, December, 1939, p. 5.

[4] Leonard Obert and Wilbur I. Duvall, "Discussion of Dynamic Methods of Testing Concrete with Suggestions for Standardization," ***Proceedings***, Am. Soc. Testing Mats., Vol. 41, p. 1053 (1941).

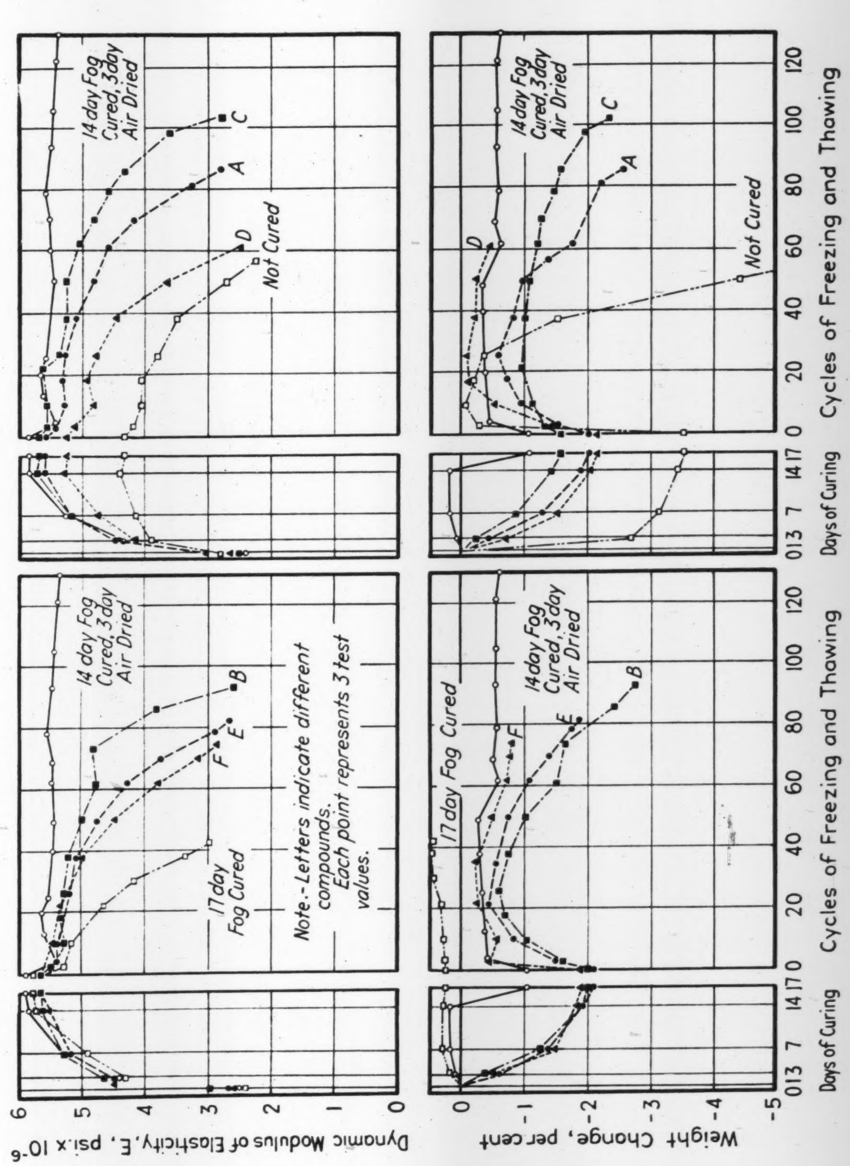

FIG. 4.—Effect of Initial Curing and Subsequent Freezing and Thawing on Modulus of Elasticity E of 4-in. Beams.

TABLE IV.—DYNAMIC MODULUS OF ELASTICITY AND WEIGHT CHANGE VALUES OF LABORATORY SPECIMENS.

NOTE.—The average value shown is the average of three tests.

Method of Curing		Curing Period					Freezing and Thawing																
		1 day	3 days	7 days	14 days	17 days	3 cycles	10 cycles	18 cycles	26 cycles	38 cycles	42 cycles	50 cycles	57 cycles	61 cycles	70 cycles	74 cycles	79 cycles	82 cycles	86 cycles	93 cycles	103 cycles	130 cycles
							DYNAMIC MODULUS OF ELASTICITY, *E*, MILLIONS PSI.																
14 days fog curing 3 days air drying	Max.	2.67	4.50	5.42	5.93	5.96	5.70		5.75	5.69	5.62		5.51		5.61	5.61		5.70			5.56	5.51	5.54
	Min.	2.52	4.25	5.17	5.66	5.74	5.37		5.50	5.41	5.37		5.37		5.40	5.41		5.46			5.42	5.42	5.26
	Avg.	2.58	4.34	5.26	5.84	5.85	5.56		5.64	5.60	5.49		5.44		5.49	5.48		5.57			5.48	5.47	5.36
17 days fog curing	Max.	2.54	4.27	4.98	5.74	5.81	5.39	5.22		4.61	3.77	3.43											
	Min.	2.46	4.21	4.96	5.70	5.75	5.32	5.16		4.29	3.02	2.77											
	Avg.	2.49	4.23	4.97	5.72	5.78	5.36	5.19		4.47	3.37	3.01											
Compound A	Max.	3.10	4.58	5.38	5.76	5.75	5.69	5.49	5.55	5.30	5.24		4.93		4.72	4.30			3.49	2.95			
	Min.	2.97	4.41	5.14	5.51	5.50	5.24	5.15	5.16	5.18	4.98		4.68		4.43	3.99			2.94	2.54			
	Avg.	3.03	4.48	5.23	5.60	5.59	5.41	5.27	5.31	5.26	5.10		4.81		4.59	4.19			3.26	2.81			
Compound B	Max.	3.09	4.65	5.41	5.80	5.85	5.57	5.49	5.46	5.47	5.41		5.30		4.97		4.94			4.12	2.92		
	Min.	2.96	4.49	5.17	5.55	5.50	5.34	5.20	5.30	5.12	5.02		4.76		4.69		4.68			3.57	2.24		
	Avg.	3.01	4.56	5.29	5.67	5.65	5.46	5.35	5.36	5.29	5.22		5.03		4.81		4.84			3.84	2.61		
Compound C	Max.	2.75	4.59	5.35	5.82	5.78	5.69	5.75	5.75	5.45	5.34		5.34		5.14	4.97		4.87		4.62	4.24	3.25	
	Min.	2.56	4.39	5.04	5.52	5.51	5.39	5.33	5.37	5.29	5.20		5.15		4.98	4.70		4.40		4.17	3.59	2.64	
	Avg.	2.66	4.47	5.22	5.71	5.68	5.56	5.56	5.60	5.35	5.25		5.24		5.02	4.83		4.59		4.34	3.90	2.81	
Compound D	Max.	2.78	4.20	4.94	5.39	5.38	5.26	4.93	5.03	4.94	4.68		4.16		3.02								
	Min.	2.57	4.10	4.61	5.17	5.16	4.93	4.70	4.75	4.52	4.21		3.17		1.92								
	Avg.	2.65	4.13	4.76	5.27	5.26	5.10	4.83	4.93	4.76	4.45		3.67		2.49								
Compound E	Max.	2.70	4.50	5.19	5.69	5.68	5.56	5.40	5.33	5.23	5.12		4.93		4.50	3.82		3.19	2.83				
	Min.	2.65	4.32	5.18	5.65	5.64	5.36	5.39	5.31	5.20	4.98		4.66		4.15	3.75		2.64	2.56				
	Avg.	2.68	4.41	5.18	5.67	5.66	5.46	5.40	5.32	5.22	5.05		4.80		4.34	3.78		2.92	2.70				
Compound F	Max.	2.67	4.70	5.44	5.83	5.71	5.70	5.70	5.56	5.47	5.26		4.96		4.80	4.45	4.24						
	Min.	2.50	4.21	5.03	5.51	5.61	5.30	5.28	5.23	5.10	4.85		4.25		3.20	2.26	2.01						
	Avg.	2.61	4.41	5.19	5.65	5.65	5.49	5.47	5.38	5.26	5.01		4.51		3.86	3.16	2.85						
No curing	Max.	2.88	4.04	4.30	4.51	4.47	4.32	4.12	4.12	4.01	3.90		3.51	2.94									
	Min.	2.71	3.79	4.03	4.24	4.19	4.05	3.95	3.85	3.49	2.85		1.98	1.81									
	Avg.	2.79	3.92	4.18	4.40	4.35	4.20	4.06	4.07	3.83	3.50		2.72	2.27									

Weight Change Values, per cent[a]

14 days fog curing 3 days air drying.	Max.	0	+0.20	+0.26	+0.22	−1.07	−0.59	−0.43	−0.42	−0.36	−0.37		−0.32		−0.60	−0.55		−0.59			−0.60	−0.59	−0.64
	Min.	0	−0.01	+0.15	+0.17	−1.03	−0.56	−0.37	−0.31	−0.27	−0.29		−0.19		−0.54	−0.44		−0.53			−0.44	−0.46	−0.51
	Avg.	0	+0.08	+0.20	+0.20	−1.05	−0.57	−0.39	−0.36	−0.32	−0.33		−0.27		−0.57	−0.50		−0.55			−0.53	−0.53	−0.59
17 days fog curing	Max.	0	+0.30	+0.36	+0.32	+0.35	+0.32	+0.42	−0.41	+0.43	+0.61	+0.53											
	Min.	0	+0.07	+0.25	+0.17	+0.16	+0.19	+0.20	−0.26	+0.22	+0.40	+0.32											
	Avg.	0	+0.19	+0.29	+0.23	+0.25	+0.25	+0.28	+0.31	+0.31	+0.48	+0.46											
Compound A	Max.	0	−0.63	−1.61	−2.23	−2.33	−1.51	−0.98	−0.86	−0.76	−0.96		−1.04					−2.49		−2.89			
	Min.	0	−0.35	−1.06	−1.74	−1.87	−1.34	−0.88	−0.56	−0.39	−0.58		−0.79					−1.83		−2.33			
	Avg.	0	−0.49	−1.34	−2.00	−2.12	−1.44	−0.91	−0.69	−0.56	−0.78		−0.95					−2.10		−2.52			
Compound B	Max.	0	−0.56	−1.56	−2.13	−2.24	−1.69	−1.09	−0.78	−0.63	−0.88		−1.21		−1.64					−2.97	−3.15		
	Min.	0	−0.42	−1.23	−1.88	−2.00	−1.57	−0.95	−0.62	−0.53	−0.61		−0.87		−1.34					−1.94	−2.11		
	Avg.	0	−0.47	−1.37	−1.98	−2.10	−1.61	−1.04	−0.70	−0.59	−0.74		−1.00		−1.51					−2.43	−2.74		
Compound C	Max.	0	−0.35	−1.00	−1.55	−1.70	−1.37	−1.10	−1.00	−1.04	−1.02		−1.08		−1.24	−1.31		−1.52		−1.57	−2.04	−2.75	
	Min.	0	−0.27	−0.92	−1.50	−1.65	−1.31	−1.10	−0.99	−1.02	−0.92		−1.00		−1.05	−1.11		−1.32		−1.44	−1.68	−2.04	
	Avg.	0	−0.32	−0.97	−1.52	−1.67	−1.34	−1.10	−1.00	−1.03	−0.98		−1.03		−1.14	−1.23		−1.43		−1.52	−1.80	−2.30	
Compound D	Max.	0	−0.87	−1.72	−2.21	−2.35	−1.40	−0.53	−0.19	−0.10	−0.21		−0.37		−0.52								
	Min.	0	−0.75	−1.54	−2.06	−2.18	−1.38	−0.46	−0.08	−0.02	−0.17		−0.20		−0.38								
	Avg.	0	−0.82	−1.63	−2.14	−2.26	−1.39	−0.50	−0.14	−0.05	−0.18		−0.26		−0.43								
Compound E	Max.	0	−0.45	−1.54	−2.18	−2.34	−1.57	−0.83		−0.48	−0.71		−0.87		−1.20	−1.57		−1.93	−2.06				
	Min.	0	−0.40	−1.44	−2.03	−2.16	−1.40	−0.80		−0.35	−0.37		−0.60		−0.94	−1.20		−1.54	−1.64				
	Avg.	0	−0.42	−1.52	−2.10	−2.25	−1.48	−0.82		−0.42	−0.54		−0.74		−1.02	−1.38		−1.74	−1.85				
Compound F	Max.	0	−0.57	−1.48	−1.98	−2.12	−1.12	−0.60	−0.42	−0.37	−0.48		−0.67		−0.93	−0.97	−1.04						
	Min.	0	−0.45	−1.19	−1.66	−1.77	−0.19	−0.43	−0.32	−0.18	−0.14		−0.32		−0.51	−0.53	−0.54						
	Avg.	0	−0.53	−1.38	−1.87	−1.99	−0.50	−0.54	−0.38	−0.28	−0.30		−0.46		−0.67	−0.74	−0.78						
No curing	Max.	0	−2.76	−3.20	−3.50	−3.56	−0.28	−0.06	−0.29	−0.40	−1.94		−5.02	−7.08									
	Min.	0	−2.56	−3.10	−3.34	−3.39	−0.22	−0.02	−0.08	−0.22	−1.23		−4.00	−6.37									
	Avg.	0	−2.63	−3.13	−3.40	−3.45	−0.25	−0.04	−0.16	−0.33	−1.49		−4.37	−6.63									

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[a] The weight change values are referred to the weights of the specimens when removed from the molds.

coupled to an expansion screw set in the wall panels. A crystal pick-up was placed on the opposite side of the panel (directly opposite the vibrator), the signal amplified through a small amplifier, and the resonant frequencies determined with high impedance earphones connected to the output of the amplifier.

RESULTS AND DISCUSSION

Laboratory Specimens:

The dynamic modulus of elasticity was selected as a criterion because it appears, after several years use in this laboratory, that of the various indexes used for indicating the quality of concrete, it yields the most dependable, uniform, and consistent results.

Figure 4 and Table IV present the average dynamic modulus and weight change values, of all groups of specimens, throughout the curing period and subsequent freezing and thawing. Weight changes (referred to the weights of the specimens upon removal from the molds) directly represent the *moisture* condition of the concrete *only* until the mortar began to spall during freezing and thawing. The commencement of spalling is indicated by decreases in the weight change values.

A comparison of the rates of decrease in dynamic modulus (Fig. 4) of the two groups of fog-cured specimens during freezing and thawing shows very strikingly the effects of varying moisture in the concrete. Those specimens which were fog cured for the entire 17-day period contained about 1.25 per cent more moisture (based on total weight of specimen) at the commencement of freezing and thawing than did the specimens which were fog cured for 14 days and then air dried for 3 days. While this may appear to be only a slight difference in initial moisture content it amounts to approximately 21 per cent of the original mixing water, and its effects, together with that of the additional moisture absorbed during freezing and thawing, are very marked, as will be seen from a comparison of the upper and lower curves of Fig. 4.

Of the compound-cured specimens those which lost the most moisture during laboratory air exposure and absorbed the most moisture during freezing and thawing showed the least resistance to freezing and thawing. For example, specimens cured with compound *C* (Fig. 4) lost 1.7 per cent of their total weight (approximately 28 per cent of the original mixing water) during curing, gained 0.7 per cent during freezing and thawing, and withstood 103 cycles; whereas, those cured with compound *D* lost 2.3 per cent of their total weight (approximately 38 per cent of the original mixing water) during curing, gained 2.2 per cent during freezing and thawing, and withstood only 61 cycles.

The no-cured specimens (Fig. 4) lost about 3.5 per cent of their total weight (approximately 57 per cent of moisture) during laboratory air exposure but absorbed an equal amount during the first few cycles of freezing and thawing, and their resistance to the durability test procedure was inferior to that of any other specimens except the group continuously fog cured until time of test. Had it not been that spalling, in the case of the no-cured specimens, commenced at about 10 cycles (thus accounting for the rapid weight loss shown in the lower portion of Fig. 4), it is probable that the weight-change curve for these specimens would have shown a continuous increase, as they undoubtedly continued to absorb water for the entire freezing-and-thawing period. On the other hand, the specimens continuously fog cured for the entire 17-day period (Fig. 4) exhibited very little or no spalling, although these specimens gained about 0.25 per cent of their original weight (or 4.1 per cent moisture in addition to the original gaging water) during this period, and continued to absorb moisture throughout the freezing-and-thawing period. The resistance of this group to the durability test procedure was even lower than was that of the group which received no curing.

TABLE V.—DESCRIPTION AND DYNAMIC MODULUS OF ELASTICITY, *E*, OF TEST WALL PANELS.

Curing Treatment	Coverage, sq. ft. per gal.	Visual Observations		Longitudinal Frequency, cycles per sec.	Dynamic Modulus of Elasticity, *E*, millions psi.
		East Vertical Surface	West Vertical Surface		
14 days water cured	...	No compound[a]	No compound[a]	3910	4.50
Compound A	165	No compound[b]	75% of surface covered with light gray compound[c]	3850	4.35
Compound B	210	Trace of light gray compound[a]	75% of surface covered with light gray compound[c]	4030	4.64
Compound B	195	No compound[a]	75% of surface covered with light gray compound[c]	3930	4.56
Compound B	195	No compound[a]	75% of surface covered with light gray compound[c]	3940	4.58
Compound C	105	75% of surface covered with light gray compound[c]	75% of surface covered with light gray compound[c]	3920	4.62
Compound C	120	75% of surface covered with light gray compound[c]	90% of surface covered with light gray compound[c]	3910	4.46
Compound C	140	90% of surface covered with light gray compound[c]	90% of surface covered with light gray compound[c]	3920	4.47
Compound C	140	50% of surface covered with light gray compound[c]	90% of surface covered with light gray compound[c]	3900	4.49
Compound D	220	Trace of brown compound[b]	50% of surface covered with brown compound[e]	3900	4.45
Compound D	290	Trace of brown compound[b]	25% of surface covered with brown compound[a]	3900	4.48
Compound D	290	10% of surface covered with brown compound[b]	90% of surface covered with brown compound[a]	4100	4.92
Compound E	300	No compound[a]	50% of surface covered with light gray compound[a]	4110	4.86
Compound E	150	No compound[a]	50% of surface covered with light gray compound[a]	3900	4.48
Compound F	300	95% of surface covered with black compound[d]	100% of surface covered with black compound[d]	3900	4.48
Compound G	250	No compound[c]	No compound[c]	3910	4.50
No curing	...	No compound[a]	No compound[c]	3830	4.33

[a] Moderate surface cracking over entire surface.
[b] Considerable surface cracking over entire surface.
[c] Surface cracking near top edge only.
[d] No noticeable surface cracking.
[e] Considerable surface cracking over top half of surface.

Figure 3 shows a comparison of the spalling of some of the fog-cured (14 days fog cured and 3 days air dried), no-cured, and representative compound-cured specimens. The fog-cured and air-dried concrete shows practically no spalling, and the compound-cured is intermediate between the fog-cured and air-dried, and the no-cured concrete. At the time that freezing and thawing of the 17-day continuously fog-cured group was discontinued (when the dynamic modulus had decreased approximately 50 per cent) the specimens exhibited no visible signs of distress, but numerous tests have demonstrated that the structural integrity of concrete may be seriously impaired before disintegration becomes visibly apparent, and it is probable that these specimens contained the numerous microscopic cracks which appear to characterize incipient disruption in concrete subjected to freezing and thawing.

Test Wall:

Results of dynamic modulus determinations and visual examination of the $3\frac{1}{2}$ yr. old test wall are given in Table V. There appears to be considerably less variation in the quality of field concrete, irrespective of the method of curing, than there is in that of laboratory concrete when the dynamic modulus is employed as an index of quality, and it seems particularly significant that after 3½ yr. the value for the panel which received no conventional curing (other than that provided by the retention of the forms in place for approximately 15 hr.) should be found to be only slightly lower than that of the panel which received 14 days of curing by means of the continuous mechanical application of water. This is probably due to the fact that the relatively large masses of concrete in the wall panels were less affected by surface evaporation during the initial curing period than were the small laboratory specimens in which the volume to exposed-area ratio was appreciably less. This also suggests the possibility that when water-tight forms remain in place for 15 hr. or more, prolonged periods of water curing may contribute less to the ultimate quality of the concrete than has been supposed, at least under the climatic conditions obtaining in the Ohio River valley at Huntington, W. Va., the location of the test wall. In this connection it may be of interest that, during the first 14 days after the wall was constructed, rainfall occurred only on the seventh day. In computing the values of dynamic modulus (Table V), no corrections were made for Poisson's ratio or for the boundaries of vibratory effect; consequently the modulus of elasticity values are somewhat lower than would be expected for the normally sound concrete of which the wall panels appear to consist.

The amounts of compound remaining on the vertical surfaces of the wall panels after 3½ yr. of outdoor exposure range from zero for compounds *A*, *B*, *E*, and *G*, to approximately 100 per cent for compound *F*. In an analysis of the data presented in Table V, it is of interest to compare the effects of weathering on the east and west surfaces of the panels. The east surfaces are exposed to the direct rays of the sun and to the wind, whereas the west surfaces, sheltered by a higher wall (Fig. 2) are exposed to the sun for only about 1 hr. each day and are almost entirely shielded from the wind. In general, this difference in the severity of exposure has resulted in greater losses of compound and in more surface cracking on exposed surfaces than on protected surfaces of the same panels.

Comparison of Laboratory and Field Test Results:

A comparison of dynamic modulus values of the panels of the 3½ yr. old test wall with those of the 17-day old laboratory specimens before freezing and thawing, is presented in Fig. 5. All wall values are arbitrarily referred to that of the 14-day "standard" water-cured panel as 100 per cent, while those of the laboratory specimens are referred to that of the fog-cured (14-day fog cured and 3-day air dried) specimens as 100 per cent. In this comparison the membrane-cured panels represented are those most closely corresponding with the 200 sq. ft. per gal. coverage (Table V) used on the laboratory specimens.

The comparative dynamic modulus values, shown in Fig. 5, of the test wall, indicate that there are no significant variations in the quality of the concrete in the wall panels after 3½ yr., regardless of the method of curing employed, while the corresponding values of the 17-day old laboratory specimens show considerable variation between fog curing and no curing, but less variation than one might expect between the various compounds used. In this connection it is interesting to note (Fig. 4), however, that even slight differences in initial dynamic modulus values of the laboratory specimens become magnified when the specimens are subjected to freezing and thawing at early ages. What effect natural weathering has produced on the modulus values of the wall panels is not known as no initial values were obtained when the wall was built due to the fact that the method of obtaining such values for concrete, *in situ*, had not been developed at that time.

CONCLUSIONS

The data presented herein indicate that *under the test conditions described*,

1. Variations in the effects of different curing methods on the relative quality of 3½-yr. old concrete wall panels exposed to the climatic conditions obtaining in the Ohio River Valley at Huntington, W. Va., are slight.

2. No relationship exists between the relative amounts of visible surface cracking, or checking of the wall panels, and the method of initial curing.

3. The moisture content (and, probably, the moisture gradient) of laboratory specimens, throughout the curing period and subsequent freezing and thawing, conditions their behavior to a marked degree.

4. A 3-day drying period, following 14 days of moist curing, significantly increases the resistance of laboratory specimens to freezing and thawing.

5. Continued absorption of moisture after casting and during freezing and thawing materially reduces the resistance of laboratory specimens to freezing and thawing.

6. The relative effectiveness of different curing compounds may be more clearly differentiated by dynamic modulus determinations during the freezing and thawing of laboratory specimens than when such determinations are made throughout the curing period only.

7. In the interpretation of laboratory test results (especially when concrete specimens are tested at such early ages as were the laboratory specimens discussed herein) extreme care must be exercised to eliminate the possibility of assuming a correlation with field behavior which may not exist.

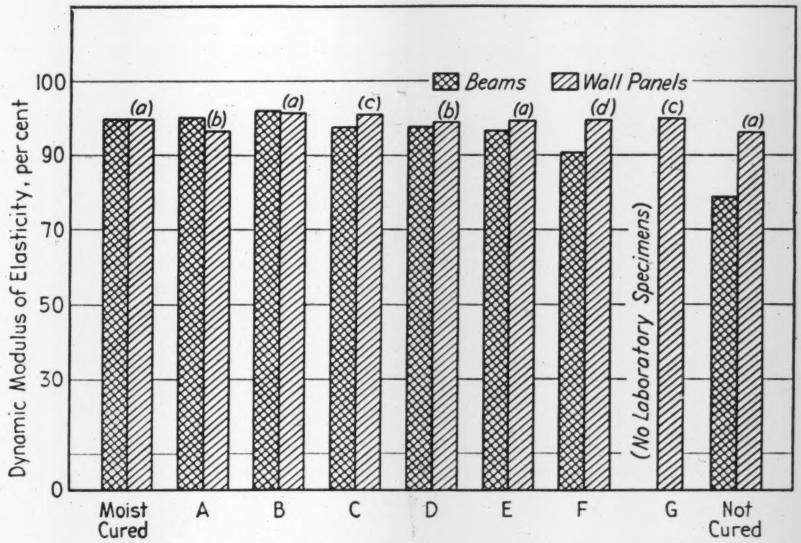

FIG. 5.—Dynamic Modulus of Elasticity of Laboratory and Field Concrete.

ADDENDUM

The authors regret that sufficient time has not been available to permit investigation of the effects of longer curing or exposure periods on the resistance of laboratory specimens to freezing and thawing. However, it is hoped that another series may be undertaken in the near future and that the results may be presented in a later paper.

It is believed that considerable promise lies in a new method of determining the dynamic modulus of elasticity of concrete, *in situ*, which is in process of development by the authors. This method consists of determining the velocity of vibration transmission through large masses of concrete over such short intervals as 1 ft. A power amplifier capable of delivering 50 w. at audiofrequencies is employed, and vibrations at different points are analyzed by means of a cathode-ray oscillograph. A reference signal from the vibrator is connected to the horizontal input of the oscillograph, and the output of a vibration pick-up is connected to the vertical input of the oscillograph. The resulting Lissajous figures represent the phase displacement of the two signals: a change from a straight line in the first and third quadrants to a straight line in the second and fourth quadrants (or *vice versa*) represents a phase change of 180 deg., a half wave length of the vibration cycle.

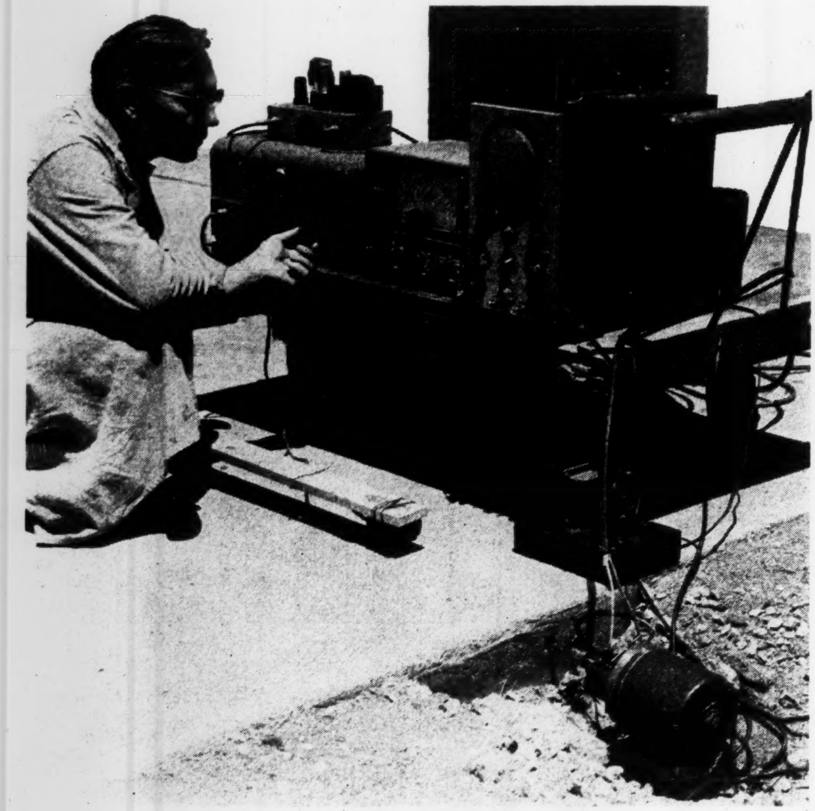

FIG. 6.—Velocity Apparatus.

The testing procedure (Fig. 6) consists of (*a*) placing the vibration pick-up at a point on any radial line from the vibrator and adjusting the frequency of vibration until the condition of either zero or 180 deg. displacement exists; (*b*) moving the pick-up along this radial line away from the source of vibration until a phase change of 180 deg. occurs; and (*c*) measuring the interval along the radial line selected, which interval is one half of the wave length of the vibration cycle. Results so obtained may be converted into terms of the dynamic modulus of elasticity in accordance with analyses developed by Lamb[5] and Timoshenko,[6] but the resulting values appear to be slightly higher than are those obtained from direct measurement of the resonant flexural frequency of small laboratory specimens of comparable concrete.

Further analytical studies will be required before the full potential value of this new method of quantitatively determining the relative quality of concrete structures such as walls, dams, highway slabs, and bridge members can be fully realized. Such studies are currently under way in the Cincinnati Testing Laboratory, and already a number of such determinations have been obtained on airfield runways in service; however, these results are not available for publication at the present time.

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[5] Horace Lamb, "On Waves in an Elastic Plate," *Proceedings*, Royal Soc. (London), Vol. 93, Series A, p. 114 (1917).

[6] S. P. Timoshenko, "On Transverse Vibrations of Bars of Uniform Cross Section," *Philosophical Magazine and Journal of Science*, Vol. 43, Series 6, p. 125 (1922).

DISCUSSION

Mr. Bailey Tremper.[1]—The authors have indicated that they intend to conduct additional experiments on membrane curing, and I should like to offer certain revisions in procedure that have been suggested by similar studies conducted by the Washington Department of Highways.

If the membranes are to be studied to determine their value in curing slabs used in runways and pavements I would suggest that the test specimens be cast and cured under circumstances similar to the intended use, that is, with the bottom surface in contact with a moist subgrade. If this is done the rating of the membrane should be considerably higher than when the specimen is coated on all sides and exposed to the air for the reason that moisture losses through the membrane will be made up, to some extent at least, by absorption from the subgrade.

Another feature of the tests as reported is that the specimens were not all brought to comparable moisture contents before starting the cycles of freezing and thawing. As the authors point out, this probably is the reason that the specimens that were cured continuously in fog did not behave as well as those that were fog cured for 14 days and then given 3 days to air dry. It is suggested that the membrane-cured specimens be brought to a moisture condition comparable to one or the other group of moist-cured specimens just prior to starting freezing and thawing. This can best be done by removing the membrane more or less completely. This should be done because the membranes, although initially moisture-repellant, cannot be depended on remaining so under the influence of weather and abrasion by traffic. Under the treatment described the durability of the membrane-cured test specimens should be more nearly in accordance with the results obtained in actual practice.

Mr. A. D. Conrow.[2]—This is my first introduction to the contents of this paper. Had I had opportunity to study it previous to this presentation, a more intelligent discussion might have been possible.

My impressions, from the results of these tests, are that one might conclude that fog curing was not a good method of curing. That is, for the development of good resistance to freezing and thawing, if loss in dynamic modulus may be considered a measure of resistance to this action.

I have had similar experiences to those indicated by these tests. We have thought, for a good many years, that immersion curing, or curing in water, might be considered to be an ideal type of curing so far as gaining the greater strength of the concrete is concerned. However, if the concrete cured in water is subjected to freezing-and-thawing alternations immediately upon removal from water, in a fully saturated condition, it will have far less resistance to that action than if it is permitted to dry out for a period before it is subjected to that action.

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[1] Materials Engineer, State of Washington, Department of Highways, Olympia, Wash.

[2] Research Engineer, Ash Grove Lime & Portland Cement Co., Chanute, Kans.

I have had some experience in that connection, when testing a concrete which had quite high compressive strength, that is, strength in the neighborhood of 4500 psi., and containing as much as 7 sacks of cement per cubic yard. When this concrete, having been in water continuously from the time of taking from the molds, was subjected to alternate freezing and thawing immediately upon removal from the water, it was disintegrated within about 30 cycles, whereas when concrete of the same composition, given as high as 60 days of fog curing, was subjected to the same treatment, it has withstood over 100 cycles without appreciable damage.

This illustrates somewhat that one may not always judge the effectiveness of curing from such tests as these.

MR. BARTLETT G. LONG.[3]—We concur in that. What we were trying to do essentially was to determine whether the dynamic modulus would give us a clear differentiation.

To my own mind, the paper has only minor significance. It does show two things, however. It shows the marked effect of the moisture condition of concrete at the time of the test, which has been brought out, and it shows the possibilities of finding out something about concrete somewhere except in the laboratory. Off and on I have been playing with concrete in the laboratory for about 20 yr., and if I have learned anything in the laboratory it is still very obscure to me. So I have come to the conclusion that if you want to know what a certain material will do in concrete, just put it in a concrete structure and go away on a fishing trip and come back in ten years and look at it and see what has happened. The new method of determining the elastic modulus of concrete in place does, I think, have promise. That is probably the only significance of the paper. I think Mr. Scholer, and dozens of others, have found out that if you saturate concrete and freeze and thaw it, it will disintegrate rapidly. The freezing-and-thawing test to me at the moment shows merely that the concrete was either wet or dry when you froze and thawed it.

There does appear to us to be one other significant point—we have not isolated it, we have not pinned it down—but there is, perhaps, a *critical* moisture condition there, the range of which, in our continuously fog-cured and in our fourteen day fog-cured and three day air-dried specimens, we have, perhaps, bracketed. There is a point there, we think—and Mr. Stanton Walker's work indicated that very strongly—there is a critical point somewhere, and if we can isolate that, pin it down, with reference to the original mixing water, then we may know more about freezing and thawing.

MESSRS. LONG AND H. J. KURTZ[4] (*authors' closure by letter*).—The points brought out by Messrs. Conrow and Tremper are well-considered and of undoubted importance.

Mr. Tremper's suggestion that field conditions be simulated, in evaluation of the method of compound curing in the laboratory, is a good one, not only in the case of curing compounds, but also in that of most other laboratory tests. In previous tests in our laboratory attempts have been made to isolate and determine the effects of subgrade moisture. However, in the present tests it was desired to compare only the moisture-retaining capacity of the various compounds, and the introduction of another variable would probably have complicated matters. In the construction work of the Corps of Engineers,

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[3] Senior Engineer, Cincinnati Testing Laboratory, War Dept., Corps of Engineers, U. S. Army, Mariemont, Ohio.

[4] Engineer, Cincinnati Testing Laboratory, War Dept., Corps of Engineers, U. S. Army, Mariemont, Ohio.

U. S. Army, concrete runways and pavements are only one item, and the yardage in buildings, dams, flood-walls and other structures is of equal or greater importance. In such construction a reservoir of curing moisture in the subgrade is not available, and the exposure of large surfaces to drying conditions imposes a severe tax on the water-retaining capacity of curing compounds. If then, certain of these materials (and an unfortunately large percentage appear to be virtually worthless) are found to inhibit evaporation from air-exposed concrete surfaces satisfactorily, it may be assumed that their use, on pavement slabs placed upon moist subgrades or sub-bases, will be found to be satisfactory also.

Referring specifically to Mr. Tremper's other suggestion that all compound-cured specimens be brought to the same moisture condition previous to commencement of freezing and thawing, it was felt that variations in the moisture content of the specimens at any given time represented the direct influence of the different compounds, and it was desired to ascertain, if possible, how this would condition the resistance of concrete, frozen at early ages, throughout the entire test procedure. Parenthetically, it is the authors' belief that one of the major advantages of the entire method of curing with compounds is the *duration* of curing effect *beyond* the 7 or 14-day period usually specified for conventional water curing. The *gradual* loss of moisture which characterizes compound curing is believed to be preferable to the sudden loss of moisture (and consequent shrinkage) which occurs when water curing is abruptly terminated (as it invariably is) the instant the specified curing period has elapsed.

Since presentation of the original paper, the authors have conducted additional tests of concrete pavement, to determine the effects of (*a*) variations in the frequency of vibration, and (*b*) in the thickness of the concrete, on the velocity of sustained wave transmission. A frequency range varying from 1000 to 2000 cycles per second has been used on slabs varying from 4 to 10 in. in thickness. The results demonstrate the consistent and reproducible phenomena of increased velocities for increases in both concrete thickness, and frequency of sustained vibration.

These tests were supplemented by a few experiments with a Shepard seismograph, in which the longitudinal velocity of the pulsation created by a single impact was measured between arbitrarily placed geophones. Although only a limited number of experiments have been conducted at this time, it appears that this latter method holds great promise provided that the apparatus can be adapted to the measurement of much shorter intervals of time than is practicable with the present equipment. Values of the modulus of elasticity, when calculated from velocities obtained by this method, are in agreement with those obtained from measurements of the natural flexural frequency of small laboratory specimens.

As this closure is written (November 17, 1943) an extensive additional investigation of the seismograph principle is planned, and it appears that with the development of much higher film-travel speeds, or preferably, the development of practical electronic circuits capable of measuring time intervals of the order of 1/10,000 sec. duration or less, the problem of accurately determining the modulus of elasticity of concrete, *in situ*, and under various conditions of stress, may be finally and satisfactorily solved.

A NEW ASPECT OF CREEP IN CONCRETE AND ITS APPLICATION TO DESIGN

BY DOUGLAS McHENRY[1]

SYNOPSIS

The nature of creep and recovery are discussed, and a principle is stated which governs the superposition of creep when the load varies with time. An equation is developed which defines creep as a function of stress, age at loading, and elapsed time after loading. This creep equation, combined with the superposition principle, provides a means for expressing the strain history when the stress history is known. The treatment indicates that problems of concrete design may be solved by the methods of conventional elasticity, provided the true loads or displacements are replaced by certain fictitious loads or displacements. Numerous laboratory tests which form the basis and the verification of the treatment are described.

The nature of the "imperfect elasticity" of concrete has been investigated in considerable detail in connection with the design and testing of concrete structures, and the present status of the investigation is described rather briefly in this paper. Although the mathematical and experimental studies have been fairly extensive, and although considerable practical use has already been made of the findings, so many questions remain still unanswered that the presentation must be considered only as an initial step toward getting laboratory data on creep into a form in which they will be useful to the designing engineer. It seems not inappropriate to mention that in spite of thirty years or more of research on creep by various investigators, very little has been published which is sufficiently fundamental to be of value to the designer of concrete structures. Some justification for presenting an incomplete investigation of the fundamentals of creep effects is provided by the present unsatisfactory condition, under which the concrete designer must resort to some such expedient as dividing the elastic modulus by 2 in order to take account of these effects. It is consoling to find that the present study indicates that this expedient of dividing by 2 is reasonably valid for a large class of problems. However, it is by no means universally valid, and it may, if used indiscriminately, result in an entirely false impression of the conditions of stress and strain in a concrete structure.

In the interests of brevity, the reader will be asked to take upon faith some statements for which the experimental or mathematical demonstration is given here inadequately or is lacking.

CREEP AND RECOVERY

When concrete is stressed to low or moderate intensity by the application of a constant sustained load, an immediate deformation occurs which is followed by further gradual deformation at a continuously diminishing rate. The terms immediate and gradual have been used only in a descriptive sense; the initial rapid deformation is not really immediate, nor can it be precisely separated from the later gradual deformation by ordinary testing technique. However, the two types of strain involved are sufficiently distinct that they may be differentiated for most practical purposes. In problems of statics, we may consider the initial rapid strain as an "instantaneous elastic strain"; the gradual strain which follows may be called "creep strain" or simply "creep." This terminology may result in some confusion, for on the basis of recent studies it is believed that creep is simply a delayed elastic deformation involving no structural changes corresponding to breakdown or slippage; however, it is convenient to distinguish between elastic and creep strains on the above basis. (An unfortunate early choice of the term "plastic flow" to describe the slow deformation of concrete has been responsible for much confusion of thought. It is suggested that use of that term be discontinued.)

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[1] Engineer, Bureau of Reclamation, U. S. Department of the Interior, Denver, Colo.

If the loaded concrete is a prism, and if the load is uniformly distributed over the ends, then the ratio of the stress to the instantaneous axial strain may be called the "instantaneous modulus of elasticity." The qualifying adjective is necessary because one or two other moduli of elasticity will be introduced later. The ratio of a constant sustained stress to the total strain (elastic plus creep) which it has produced at any time will be called the "sustained modulus of elasticity" (or the "sustained modulus") at that time. This modulus will probably reduce in value continuously as long as the load is maintained (measurements up to 10 yr. have been reported (**1**)),[2] but the rate of change diminishes in such a way that a limiting value is approached as time goes on. Usually one half of the ultimate creep takes place in the first 5 to 60 days after loading.

It will be presumed here that the sustained modulus is independent of the magnitude or the sense of the stress within a range of stress which excludes any close approach to failure. This is mildly contradicted by some (not all) laboratory tests; but the contradiction is not particularly serious, in fact, it appears to be much less serious than are the difficulties encountered in following any other presumption—if any other suitable generalization could be found, which is questionable.[3]

Returning to the loaded prism, when the load is removed, two types of axial recovery occur. Both will be treated as manifestations of elastic recovery, but as in the case of the initial deformation, they will be distinguished by calling them elastic and creep recovery. The instantaneous axial recovery (called the elastic recovery) is given by the quotient of amount of stress removed divided by instantaneous modulus of elasticity at the time of removal. The quantitative aspect of the gradual creep recovery is slightly more subtle and will be discussed in some detail. This gradual recovery has been accorded scant attention by most observers; but it is a most interesting phenomenon, and it provides a splendid key to the somewhat cryptic relationship between variable stress and corresponding strain in concrete.

The author's attention was first directed toward creep recovery when measurements of strain and deflection in concrete dams indicated that creep occurred about equally for positive and negative load increments; that is, that creep and recovery were about equal for equal, but opposite, load changes. This was considered at the time (1938) to be contrary to laboratory data and to widely accepted opinion which cited recovery as 10 to 20 per cent of the creep. But recovery of a very much larger percentage was indicated beyond question by the structural measurements; so an apparent impasse developed between the laboratory and the structure. It seems now, as frequently happens when a better understanding is reached, that both sides were right. Further clues were provided by a series of creep and recovery measurements published by Theuer (2) involving tests of concrete at various degrees of saturation and various temperatures. The creep curves displayed a variety of shapes from smooth, gradual climbs to steep ascents with sharp breaks. Although it was not mentioned by the experimenter, the curves of creep and recovery for each test were so remarkably alike in shape that it was apparent that the relationship between the two was much more than haphazard.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1084.

[3] This does not necessarily conflict with various other treatments of creep which assume some form of nonlinear stress-creep relationship. It is recognized that some problems, especially those involving high stress, may require investigation of the effects of a deviation from linearity.

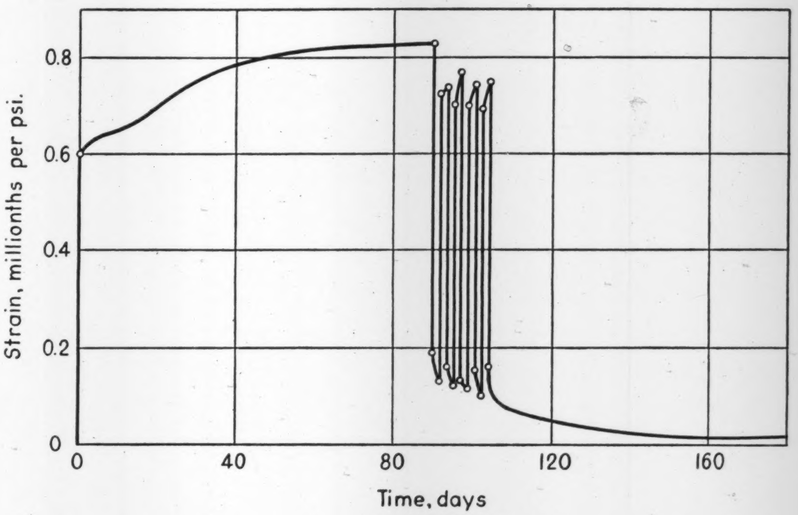

FIG. 1.—Total Strain and Recovery in Rock Core.
Loaded for 90 days, then subjected to four cyclic loadings, and finally unloaded at age of 104 days.

PRINCIPLE OF SUPERPOSITION

Search for a quantitative description of this relationship lead to the hypothesis of a principle of superposition of time effects in concrete, expressed thus:

> The strains produced in concrete at any time t by a stress increment applied at any time t_o are independent of the effects of any stress applied either earlier or later than t_o. The stress increment may be either positive or negative, but stresses which approach the ultimate strength are excluded.[4]

Any appearance of simple finality in this statement is deceptive; for if the statement is verified it may, in combination with the presumption that the sustained modulus is independent of stress intensity, constitute the beginning of a rather extensive analysis of the stress-strain relationship in concrete. The principle has been verified to a reasonable degree of accuracy for certain special, simplified cases. Complete verification, together with a definition of the stress limits within which it applies, must await further tests, particularly tests under triaxial loading.

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[4] This idea dates back at least to 1870 when Boltzman (3) investigated the mathematics of "elastische Nachwirkung." Boltzman's work has recently been revived, particularly by Leaderman (4) in connection with textile fibers and plastics.

An immediate inference from the principle is that when a sustained load is removed from concrete which was first loaded when so well aged that the properties had become constant, the load removal will be followed by an eventual complete recovery of both the instantaneous and the creep strains. This is because a load removal may be represented by the addition of an equal load of opposite sign. If the two opposing loads are imagined to be maintained indefinitely, it is apparent that the two opposing components which determine the creep recovery must asymptotically approach equality and thus will cancel each other eventually. A little consideration will show that the curves of creep and recovery will not be identical, even for very old concrete, unless the load has been sustained for such a long time that the rate of creep has reduced to a negligible amount. This is in qualitative accord with the measurements on service structures, mentioned earlier. Laboratory tests of creep and recovery in concrete so old that the properties are stable are apparently not available; but some types of rock conform to the principle. Figure 1 shows the elastic and creep deformations and recoveries for a specimen of volcanic agglomerate subjected to a sustained load of 400 psi. for 90 days, followed by four later applications of the load. The 90-day loading produced a creep strain of 91 millionths; all but 6 millionths of the strain was recovered during 80 days following the final load removal.

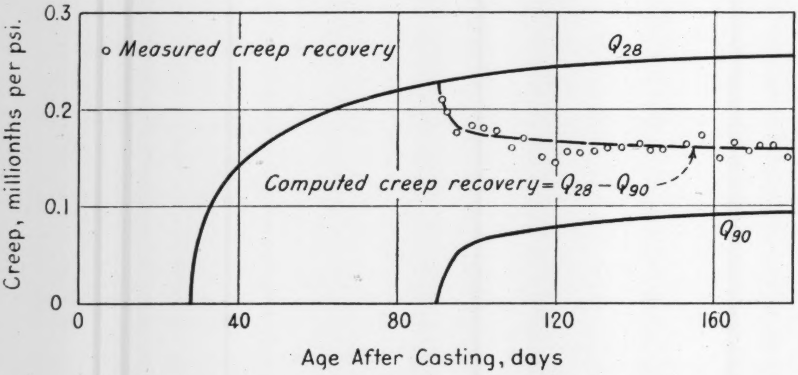

FIG. 2.—Creep and Creep Recovery in Concrete Cylinder.

Q_{28} and Q_{90} show the creep following loading at ages of 28 and 90 days, respectively. The curve of recovery following unloading at age of 90 days is given by subtracting Q_{90} from Q_{28}.

In the usual creep tests on concrete, the load is applied at a rather early age, say 28 days; so that in investigating superposition of time effects the subsequent change in properties of the concrete cannot be neglected. Figure 2 shows the results of a laboratory test in which the recovery following unloading at age 90 days was measured and was also computed from the creep curves by the above principle. The recovery was computed simply by subtracting the creep curve for loading applied at age 90 days from the creep curve for loading applied at age 28 days.[5] The computation of recovery constitutes a simple as well as a severe test of the principle. Tests involving continuously varying stress and strain come somewhat closer to most service conditions, and such tests will be described later.

THE CREEP EQUATION

It will be noted from the principle of superposition that the state of strain of concrete at any time is a function not only of the forces acting at that time, but of the entire past stress history. Some very important problems in concrete construction have to do with the development of stress due to the early application of weight forces or temperature strain. In all such problems it is necessary that the elastic and creep properties of the material be known continuously from the time it first acquires an ability to sustain stress. Determination of the continuously changing instantaneous modulus of elasticity is a relatively simple laboratory procedure. Measurement of the creep characteristics is more involved, especially if these must be determined for some particular condition of temperature variation or of drying. About the most we can expect from any reasonable laboratory program is to determine the creep curves for loads applied at two or three different ages, say 7, 28, and 90 days. The complete problem of the stress-strain relation requires that such laboratory tests be interpolated and extrapolated to yield a complete family of creep curves, or a creep surface having as the variables creep, age at loading, and elapsed time after loading. Construction of such a surface obviously calls for an equation involving coefficients determined to fit the particular laboratory data, and the requirements are such that this equation cannot be a particularly simple one. Interpolation or extrapolation by a purely empirical equation is usually uncertain; so it is desirable that the equation should have at least some pretense to a rational basis.

As a first approach, it will be presumed that when a constant sustained load is applied to concrete of stable structure and properties, a certain amount of potential creep is introduced and that this creep is manifested in accordance with the law of mass action; that is, that the rate of creep is at all times proportional to the amount of potential creep remaining. This simple condition is expressible as $\alpha(1 - e^{-rt})$, where t is time after loading, α and r are constants, and e is the base of natural logarithms. It will also be presumed that during the period before the properties are stabilized a transient creep property exists, disappearing at a rate proportional to itself, and that creep due to this disappearing property is likewise governed by mass action and may be added to that due to the stable property. By postulating various types of internal behavior under load, and various changes in structure as the concrete ages, equations of almost unlimited complexity might be built up; but there appears to be little reason at this time for adopting any but the simplest form. The following is suggested as tentatively satisfactory:

$$Q = \alpha(1 - e^{-rt}) + \beta e^{-pK}(1 - e^{-mt}) \quad (1)$$

where:

Q = the creep due to unit load,
K = age at time of loading,
t = time after loading, and
α, β, r, p, m = constants which must be determined from laboratory tests.

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[5] Figure 2 is from a series of tests in which 26 combinations of loading and unloading were investigated. All specimens were sealed in copper containers. One group was held at constant temperature, and another group followed a typical mass concrete-temperature cycle. The agreement in measured and computed recoveries indicated that superposition is valid as a working principle, but the average agreement was somewhat less satisfactory than that shown by the figure, indicating (as would be expected when dealing with a material which is notorious for its defiance of all simple rules) that 100 per cent precision should not be assigned to the superposition principle.

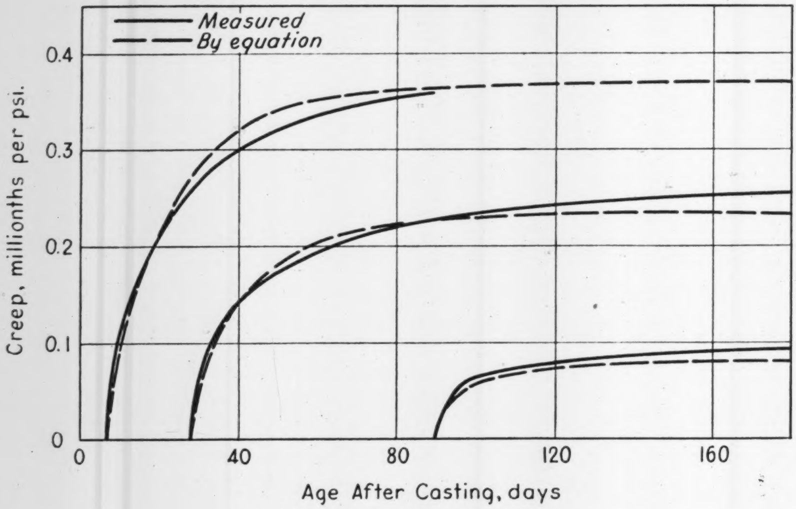

FIG. 3.—Creep Curves.
Shasta Dam concrete in sealed 6-in diameter cylinders

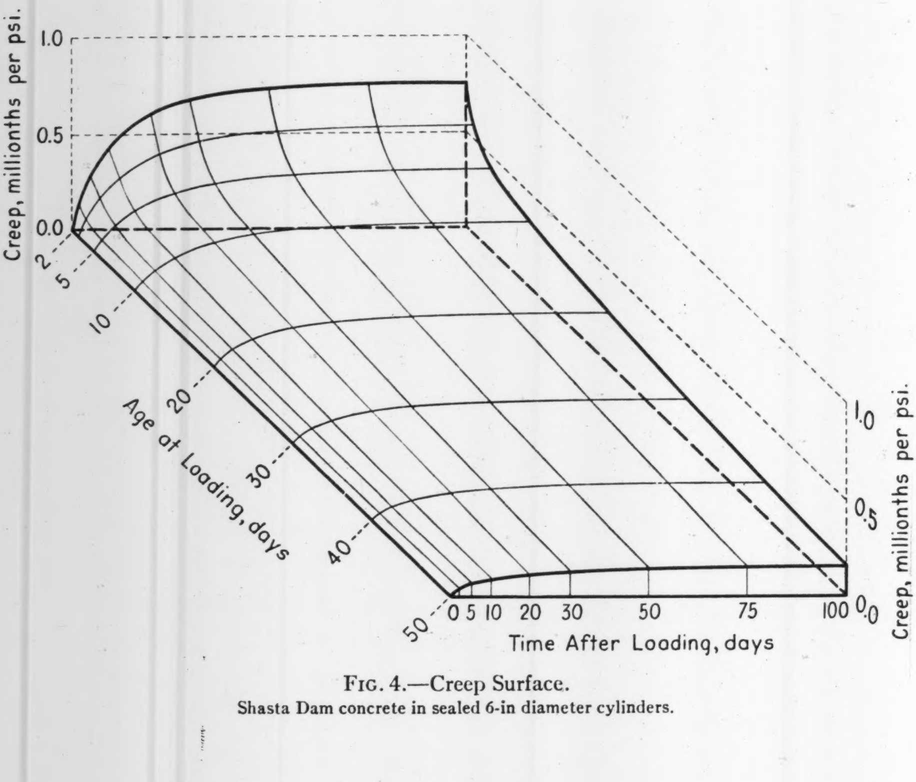

FIG. 4.—Creep Surface.
Shasta Dam concrete in sealed 6-in diameter cylinders.

Although five constants are involved, their determination by trial is not difficult.[6] At least three laboratory curves should be available for the purpose, preferably representing loadings at ages 7, 28, and 90 days.

It will be noted that the equation defines a creep curve for the case when K (age at loading) is equal to zero. This means that age must be reckoned from some time after casting, when the concrete has begun to develop definite elastic and creep properties. One half day after casting appears to be a suitable choice for zero age, but this point requires further investigation. The equation is not wholly acceptable, for it appears to be unsatisfactory when the loading age is less than about 5 days and is inaccurate, in some cases, when the duration of loading exceeds about 1 yr. Certain variables, such as temperature or extent of drying, may require consideration in particular problems; but it may be assumed that the creep curves have been measured in accordance with the conditions of the problem, or else that a method is available for making a conversion to these conditions.

In Fig. 3 are shown the laboratory creep curves for sealed 6-in. diameter cylinders made from Shasta Dam cement and aggregate loaded at ages 7, 28, and 90 days, together with the creep curves given by the equation for a load of 1 psi.

$$Q = 0.04(1 - e^{-0.60t}) + 0.398e^{-0.025K}(1 - e^{-0.06t})$$

where t and K are in days and the unit for Q is 1 millionth. Figure 4 shows a portion of the surface defined by the equation. This equation did not fit the observed curves satisfactorily for loading at ages earlier than 5 days, but its accuracy was extended to cover 2-day loading by adding a third term $1.55e^{-0.726K}(1 - e^{-0.122t})$.

Accepting the principle of superposition, Eq. 1 provides a means for expressing the entire strain history of a concrete specimen or member subjected to axial loading when the stress history is known. Simple graphic or arithmetic procedures may be devised for applying the equation, or rigorous solutions may be developed for particular cases. If the varying stress has an initial value $\sigma = 0$ when $K = \tau$ and varies continuously from $K = \tau$ to $K = T$, the creep will be[7]

$$\bar{Q} = \int_{\tau}^{T} \alpha(1 - e^{-r(T-K)}) \frac{d\sigma}{dK}\, dK + \int_{\tau}^{T} \beta e^{-pK}(1 - e^{-m(T-K)}) \frac{d\sigma}{dK}\, dK \quad (2)$$

where $\bar{Q}$ indicates creep due to the total stress. Equation 2 may be integrated for stress variations corresponding to logarithmic curves, sine waves, or any other elementary function. For a load varying at a constant rate, for example, $\frac{d\sigma}{dK} = \text{constant} = V$, and

$$\bar{Q} = V\alpha\left[T - \tau - \frac{1}{r}(1 - e^{-r(T-\tau)})\right] + V\beta\left[\frac{1}{p}(e^{-p\tau} - e^{-pT}) + \frac{1}{p-m}(e^{-pT} - e^{-p\tau-m(T-\tau)})\right] \quad (3)$$

Rigorous procedures might also be applied to the integration of the instantaneous elastic deformations; but approximate arithmetic methods are so simple for this part of the problem that it seems preferable to use them. It will

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[6] In some cases, Eq. 1 may be simplified by making r and m equal, giving

$$Q = (\alpha + \beta e^{-pK})(1 - e^{-rt})$$

[7] Lorman (5) and Ehrenberg have used a similar treatment for the following special case: creep expressed by a hyperbolic equation, no change in properties, and superposition restricted to continously increasing loads.

be shown later, too, that by a slight variation of the above procedure the need for integration of the instantaneous elastic strains may be avoided.

It should be emphasized that the stress-creep relationship as developed here is intended to be entirely general in so far as it is independent of the source of the stress—whether from load, temperature change, drying shrinkage, or other cause. In the application it will be required, of course, that the creep coefficients be known for the particular conditions of temperature or moisture content specified by the problem. If the problem is one in which the stress is known at a particular point in a structure, the procedure here outlined will give the corresponding strain; to this must be added, to give the total deformation at the point, the effects of *unrestrained* deformation due to temperature change or whatever other source of volume change may be present. If the total deformation at the point is the given quantity, then the values of *unrestrained* deformations due to all causes other than stress must be subtracted before converting the strain into stress. When over-all behavior, rather than conditions at a point, are in question, then resort may be had to methods described later in this paper.

When the varying strain is the given quantity, a rigorous treatment requires that Eq. 2, with an added term to express the integral of the instantaneous elastic deformations, be solved as an integral equation. This solution has not been developed in a very satisfactory form, but a fairly simple step-by-step procedure has been devised which in the limit, as the steps are taken smaller, converges to the exact solution.[8]

In case the stress is constant, the sustained modulus of elasticity as defined previously is equal to $\frac{E_\tau}{(1 + QE_\tau)}$ where E_τ is the instantaneous modulus at the time of loading. For the case of varying stress, the term "sustained modulus" loses its meaning; in fact, the ratio of stress to strain has in general little meaning if the stress varies, for this ratio may range from $-\infty$ through zero to $+\infty$. It is convenient, however, to have a name for the ratio when the stress is variable, and the expression "effective modulus of elasticity" seems to be as good as any. The concept of an effective modulus has some utility, for its values may be determined for certain types or patterns of stress variation and then applied as an approximation to relate stress and strain for other similar patterns. It is found, for example, that if the stresses change very slowly with time, as in the slow cooling of a large mass of concrete, or the gradual filling of the reservoir behind a large dam, then the effective modulus will not differ greatly from the sustained modulus. Therein is found the justification for the use of a reduced value of the instantaneous modulus in a large class of design problems.

It is an unfortunate circumstance that the creep of concrete is both an important factor and a difficult one to handle. Perhaps the most impressive evidence of the importance of creep is presented by tensile failures occurring in concrete when the strains are compressive, the ratio of stress to strain being negative. This type of failure has often been produced in the laboratory (see Fig. 5, for example), and the author has frequently suspected a similar condition in service structures. Design methods in which creep is introduced through the use of a reduced value of the elastic modulus are obviously not adapted to handle such situations. It is believed that an approach along lines similar to those given here is prerequisite to the development of any rational treatment of stresses and strains in concrete. If the treatment should prove to be fundamentally sound, regardless of its complexity, the necessary simplifications for special cases will be forthcoming eventually.

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[8] See Appendix, p. 1084.

The above hypotheses concerning the behavior of concrete under load have been investigated with some degree of thoroughness by a review of all published creep tests readily available, and by additional laboratory tests not previously reported. In spite of this investigation the author is not yet prepared to assign any specific limits of accuracy to the principle of superposition of time affects or to the assumed linear stress-creep relationship, nor to ascribe to these any virtues beyond those of reasonable working bases. It is almost axiomatic that when dealing with concrete any thought of high precision must be dropped at the outset. Previously published data indicated that when aging was taken into account, creep recovery amounted to 60 to 100 per cent of that expected from superposition;[9] the additional laboratory tests, which are perhaps better indicators because they were designed with the particular purpose in mind, all showed recoveries either equal to or slightly greater than those computed by superposition.

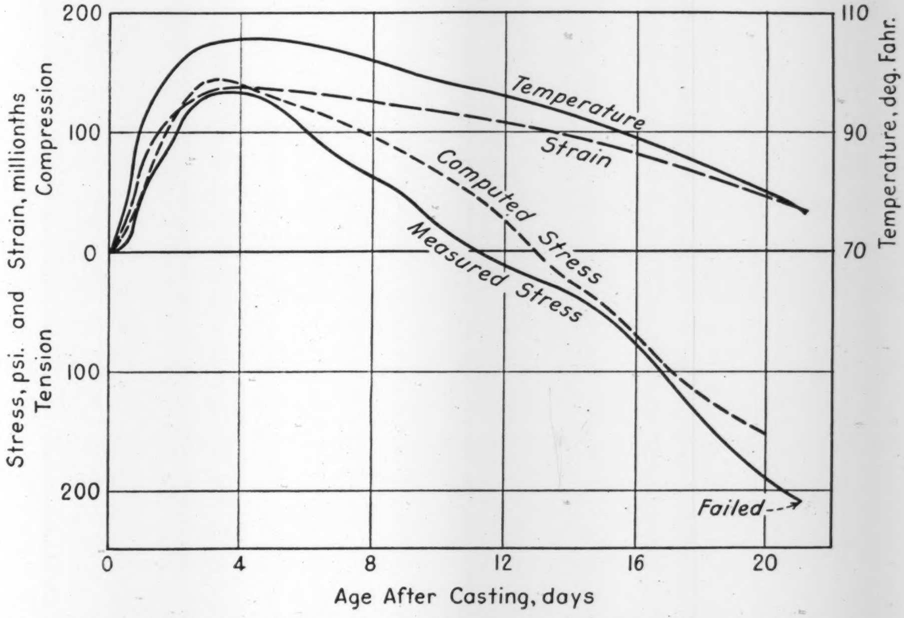

FIG. 5.—Stress in Concrete Cylinder Held at Constant Length During a Temperature Cycle.

VARIABLE LOAD TESTS

To test the accuracy of Eq. 1 and 2 when the stress and strain are continuously varying, eight laboratory tests involving three different concretes were used. In each case the constants of Eq. 1 were determined from constant load tests and the creep surface was plotted. This surface was then used for numerical solution of Eq. 2 by the step-by-step process which was mentioned. In each case of variable loading, the measured strains were taken as the known quantities, and the stresses computed from these were compared with the measured stresses. In the test shown by Fig. 5, a cylinder of concrete was held at constant length while undergoing a temperature change typical of the initial rapid temperature rise and the gradual subsequent decline encountered in many types of concrete construction (6). The constant length was maintained by applying measured axial loads in compression or in tension, as required. The strain is taken as the difference in unit elongation of the stressed specimens and of stress-free control cylinders following the same temperature changes. Modulus of elasticity was determined from companion specimens tested at frequent intervals, starting at age 0.5 day. All the cylinders were sealed in light copper jackets to prevent any loss or gain of moisture. The constant-length specimen failed when the tensile stress reached 212 psi. at age 21.25 days, when the temperature was 6 deg. higher than the starting temperature and the strain was 45 millionths in compression. The measured and computed stresses are in fair agreement throughout the entire period. Figure 6 shows the results of a similar test, using a different concrete and covering a longer period of time (81 days). Again, the measured and computed stresses are in reasonable agreement throughout the period.

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[9] The figures given are not too reliable, for most of the tests required that certain supplemental data be supplied by estimates which may or may not have been reasonably good.

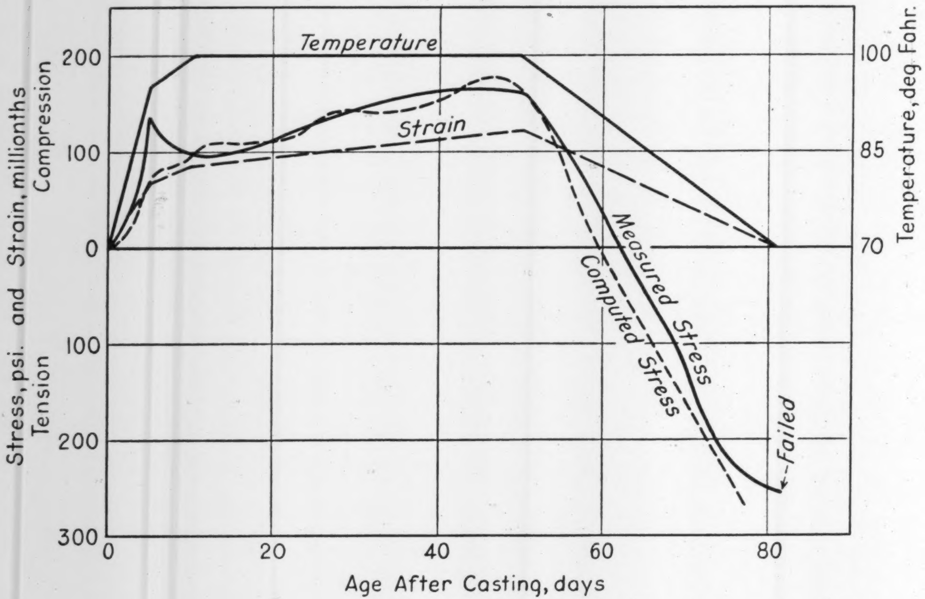

FIG. 6.—Stress in Concrete Cylinder Held at Constant Length During a Temperature Cycle.

CREEP AND SUPERPOSITION IN SYNTHETIC RESINS

It is interesting—and significant—to note that the stress-strain relationship in concrete is not an anomaly in nature, but that certain other materials, notably synthetic resins, show the same type of relationship. It may well be that this similarity in behavior results from a degree of similarity in internal structure. Neither in the case of cement paste nor of the resins is the internal structure or the mechanism of creep understood in full detail; but it is consistent with known facts to imagine a mixed solid-viscous phase which exhibits a sort of dash-pot action accounting for the delayed elasticity in each case. Both the cement paste and the resin harden by gelation, forming in the first case a heterogel in which free molecules of one material (water) are restricted in their movements by a solid structure of different material, and in the second case an isogel in which the free and the fixed molecules are alike. In the cement paste it appears that the internal reactions which support a rapidly applied load are at first supplied by the combined structure, but the fluid gradually relieves itself of stress by migrating within the system, thus transferring more and more of the load to the solid phase. When the load is removed, the immediate deformations produce a new configuration which again stresses the fluid, starting it on a new migration and hindering the recovery in the same way that the original deformation was hindered. In the resins and similar materials the distinction is usually made between tight and loose molecular bonds rather than between solid and fluid states, and the concept of fluid migration is replaced by one which involves gradual breakage and subsequent reforming of the secondary (loose) bonds.

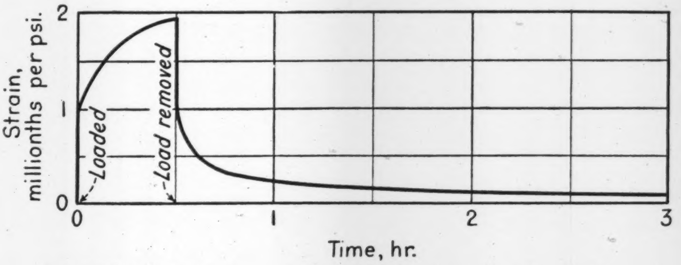

FIG. 7.—Total Strain and Recovery in Cast Phenolic Resin.

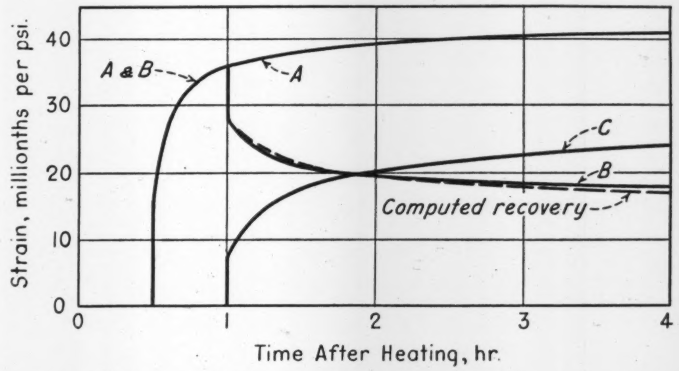

FIG. 8.—Measured and Computed Recovery in Cast Phenolic Resin After Heating. Specimens heated at 100 C. for 10 min. before testing.

The curves of elastic and creep deformation for a specimen of Catalin (thermosetting phenol-formaldehyde) are shown by Fig. 7. The behavior is generally identical with that which is here postulated for completely aged concrete, approaching complete recovery asymptotically. It was also found that the aging effect in concrete could be simulated in this material. When Catalin is heated at 100 C. for 10 min., the elastic modulus is reduced by about three fourths and the creep is correspondingly increased; the original properties are then recovered gradually over a period of 2 or 3 days. A 1-day test of the resin, then, will simulate a concrete test requiring a year or more for completion. A typical result from this type of test is shown by Fig. 8. Specimens A, B, and C were each heated for 10 min. at 100 C. and were subsequently loaded according to the following schedule, using the same load in each case: A was aged for 30 min., loaded, and the load sustained for 3½ hr.; B was aged for 30 min., loaded, and then unloaded 30 min. later; C was aged for 60 min., loaded, and the load sustained for 3 hr. If the principle of superposition is applicable, the recovery shown by specimen B should be equal to the difference of the deformation curves for specimens A and C when plotted in their proper positions along the time scale. The diagram indicates this to be the case almost exactly.

EXTENSION TO THREE DIMENSIONS

The generalized three-dimensional treatment of the preceding concepts has been worked out in considerable detail, but it will be covered only briefly here.

In discussing three dimensions instead of one, it seems preferable to simplify the mathematical treatment by introducing a somewhat more complicated (although perhaps more logical) definition of creep. We know that as concrete ages its instantaneous modulus of elasticity increases. This may be observed readily in the laboratory by loading a concrete cylinder axially at successively greater ages, removing the load each time as soon as the strains have been measured. If the load is the same for each test, the elastic strains will be progressively smaller. If the load is maintained continuously instead of being removed after each test, it is reasonable to conclude that the same thing happens, that is, that the elastic strain progressively diminishes. Referring to Fig. 9, the creep which was formerly designated Q and measured from a constant ordinate now becomes the quantity Q' measured from the continuously decreasing ordinate which is equal to $1/E$. (Attention was called to this by C. S. Whitney in 1932 (7).) It is really immaterial to the present purpose whether this conclusion is right or wrong; the final answer will be the same in either case. However, its acceptance permits some simplification, because now the elastic part of the strain produced by a variable stress is at all times equal simply to the quotient $\frac{\sigma}{E_T}$, whereas by the former definition it would be expressed by an integral. If it were desirable to name the two creeps

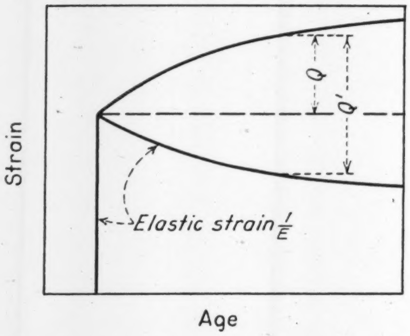

FIG. 9.—Apparent Creep, Q, and True Creep, Q'.

the first, Q, might be called the "apparent creep" and Q' the "true creep." The remainder of this discussion concerns the latter.

The exponential creep equation, Eq. 1, is probably just as satisfactory for expressing Q' as for expressing Q; but in developing a set of general equations the question of the creep equation may be left open, expressing Q' only as a function of age at loading, K, and of elapsed time, after loading, $T - K$. Thus, for a constant unilateral stress σ_o:

$$\epsilon_T = \frac{\sigma_o}{E_T} + \sigma_o Q' = \frac{\sigma_o}{E_T} + \sigma_o f(K, T - K) \quad \text{..(4)}$$

and for a varying unilateral stress, σ, having the value of $\sigma = 0$ when $K = \tau$,

$$\epsilon_T = \frac{\sigma_T}{E_T} + \int_\tau^T f(K, T - K) \frac{d\sigma}{dK} dK \quad \text{...(5)}$$

The general problem of stress and displacement in concrete, granting the validity of all that has preceded, may now be treated by a method which reduces the problem to one in conventional elasticity; that is, if the design problem can be solved for a perfectly elastic material, it can also be solved for concrete.

Consider first a one-dimensional case: a prism loaded axially by uniform stress σ with $\sigma = 0$ when $K = \tau$ and varying continuously until $K = T$. The total axial strain at age T is given by Eq. 5. It is evident from a little consideration of this equation that if the material exhibited no creep but showed only the instantaneous reactions to the load variations (corresponding to the instantaneous modulus E_T), then the same strains would be produced if the load intensity at time T instead of being σ_T were equal to σ'_T where

$$\sigma'_T = \sigma_T + E_T \int_\tau^T f(K, T - K) \frac{d\sigma}{dK} dK \quad \text{..(6)}$$

For this simple case, then, the strains and displacements in the prism may be found by treating the problem as one involving only instantaneous elastic action (so-called "perfect elasticity"), using σ' instead of σ as the intensity of the end loads. If the load were applied instantaneously instead of gradually, then Eq. 4 would be used in place of Eq. 5. If a reasonable assumption is made regarding Poisson's ratio,[10] then a treatment which is identical with that followed in deriving the fundamental equations of conventional elasticity shows that this method of adding a fictitious load to the real load is not restricted to simple cases but is equally valid for two- and three-dimensional cases and for nonuniform stress distributions. Body forces, such as dead-weight loads, may also be included by the same method. The deflection of an unrestrained plain concrete beam under any type of loading, for example, may be computed by the usual design formulas by supposing that both its weight and the external loads differ from their true values by amounts which depend upon the age of the concrete, the nature of the load variations with time, and the creep properties. This treatment will yield the strains and displacements, but the stresses will be determined by the true rather than the fictitious loads and will be unaffected by the creep or by the changing properties of the concrete.

A conclusion of some importance is that in any case of conventional elasticity in which the stresses are independent of the value of E, the same stresses will hold if the material is concerte. This applies even though the loads vary with time and though the concrete is aging, provided the aging affects the properties uniformly throughout. In such cases, then, regions which are more highly stressed than others are not relieved by a redistribution of stress due to creep. (This statement will not apply if the stresses are higher than those to which this study is restricted.) It will be noted, of course, that in all cases of reinforced concrete, or of temperature or of drying stress, the stress depends on the elastic constants, and creep may therefore be effective in relieving stress.

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[10] It is assumed that Poisson's ratio is the same for creep and elastic strains, and that it is independent of age.

In a large class of problems the specified conditions are given not as loads but as displacements. This type of problem yields to the same sort of treatment, although considerable mathematical difficulty is encountered if the creep function is retained as given previously. It is expedient for this case to introduce a new function which we may call the "stress-relaxation function," R'. If a prism of concrete is loaded axially and then maintained at constant strain, the load required to hold this strain will gradually diminish, approaching asymptotically to some ultimate constant value. As in the case of the "true creep," we may presume that if relaxation were absent the stress would at all times be given by the product $\epsilon_T E_T$.

Denoting the stress relaxation by

$$R' = \phi(K, T - K) \text{..........} (7)$$

we have for constant strain ϵ_o,

$$\sigma = \epsilon_o E_T + \epsilon_o \phi(K, T - K) \text{.....} (8)$$

and for a variable strain (with $\epsilon = 0$ when $K = \tau$),

$$\sigma = \epsilon_T E_T + \int_\tau^T \phi(K, T - K) \frac{d\epsilon}{dK} dK \text{..} (9)$$

If, then, in the axially loaded prism the varying strains are given and we wish to find the corresponding stress, we may treat the prism as though it were without creep, of elastic modulus E_T, and subjected to the strains ϵ' where

$$\epsilon' = \epsilon_T + \frac{1}{E_T} \int_\tau^T \phi(K, T - K) \frac{d\epsilon}{dK} dK \text{...} (10)$$

Instantaneous strain changes would require using Eq. 8 instead of Eq. 9. An extended analysis shows that the generalization of this method of using the relaxation function is entirely valid for more complicated cases.

Summarizing the above generalized treatment, it appears that in a large class of problems (those in which, by conventional design methods, only the loads and the external dimensions are needed to solve for the stresses) the stresses are not influenced by creep nor by changes in the elastic or creep properties of the concrete. The strains and displacements are of course influenced by these properties, and such influence is the same as that of adding fictitious loads which vary with time in a manner depending upon the changing properties of the concrete and the nature of the time variations of the true loads. If the problem is one in which displacements or strains rather than loads are specified (as in the case of strain measurements, or of shrinkage with a known degree of restraint), then certain fictitious displacements similarly determined may be used to find the stresses by the usual methods applicable to perfectly elastic materials. The nature of these fictitious displacements is such that they will ordinarily indicate a reduction in the magnitude of the stresses.

A more difficult class of problems in which the displacements are specified in terms which involve the stresses (as in reinforced concrete, or a concrete structure on an elastic foundation) has not been discussed, for the difficulties inherent in treating this class have not been satisfactorily overcome.

Acknowledgments:

The author desires to acknowledge the assistance of W. H. Jurney, particularly in the development of the general treatment of three dimensions. The work is being conducted in connection with the

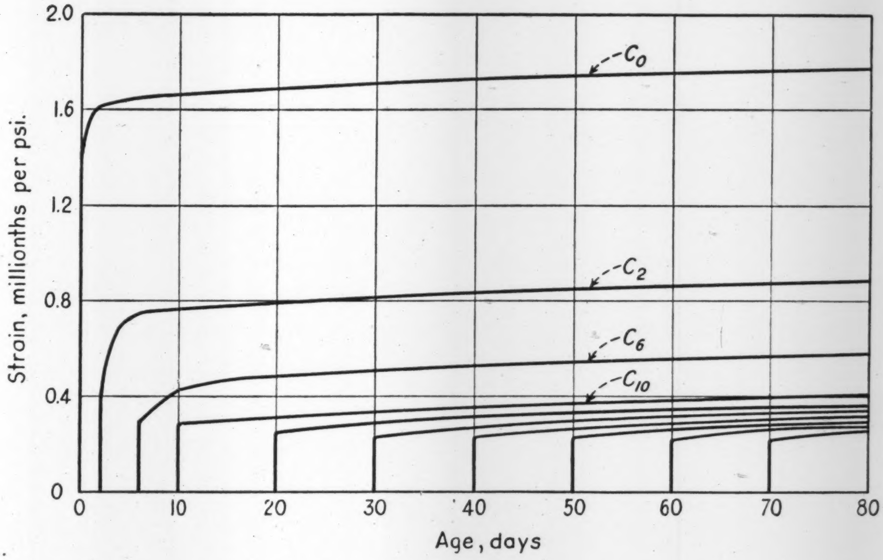

Fig. 10.—Creep Curves for Concrete.

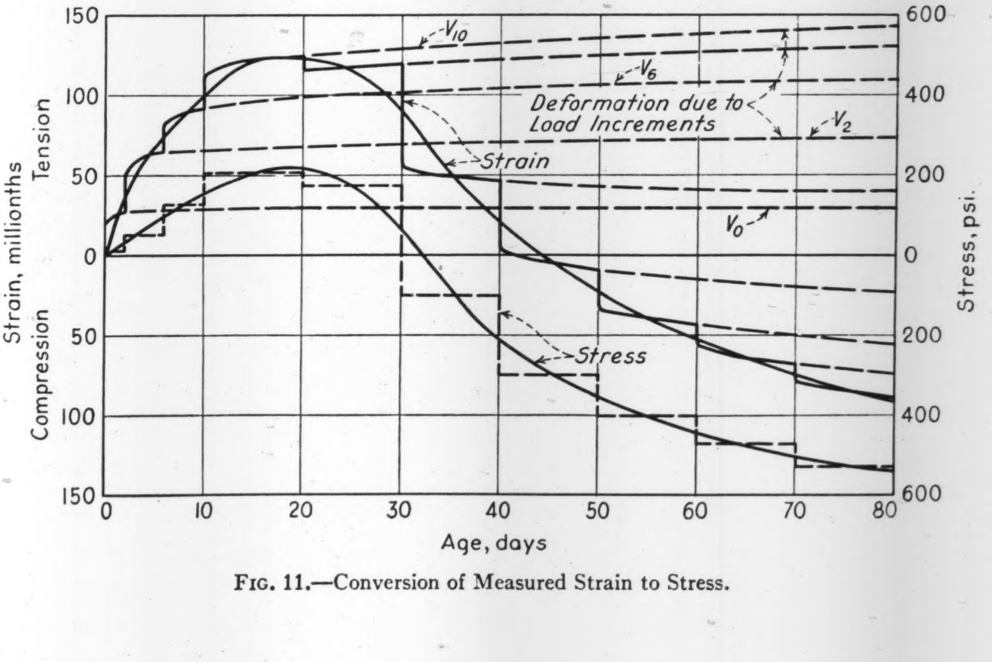

Fig. 11.—Conversion of Measured Strain to Stress.

design and testing of structures of the Bureau of Reclamation under the immediate direction of K. B. Keener, Designing Engineer on dams, R. F. Blanks, Materials and Testing Engineer, and J. L. Savage, Chief Designing Engineer. The Bureau of Reclamation is headed by Commissioner John C. Page, Washington, D. C., and all engineering and construction work is directed by S. O. Harper, Chief Engineer, with Headquarters in Denver, Colorado.

APPENDIX

CONVERSION OF STRAIN TO STRESS

The conversion of strain into stress may be effected directly by the stress-relaxation function; however, because little is known about this function it will usually be required that the conversion be made by means of the creep function. This may be accomplished by a step-by-step procedure which, in the limit, as the steps are taken smaller, converges to the exact solution. The method is illustrated by Figs. 10 and 11.

Let C_0, C_2, C_6 .... designate the curves so marked on Fig. 10; that is, the curves of total deformation for a constant sustained load of unit intensity applied at ages 0, 2, 6, ... days. Let V_0, V_2, V_6, ... designate the curves so marked on Fig. 11, representing deformation due to the cumulation of certain stress increments which are designated S_0, S_2, S_6,.... To determine curve V_0 we use $V_0 = S_0 \cdot C_0$, where S_0 is so chosen that the value of V_0 at age 1 day (the mid-point of the first time interval) is equal to the given strain at age 1 day. Curve V_2 is then obtained by using $V_2 = V_0 + S_2 \cdot C_2$ with S_2 so chosen that the value of V_2 at the next mid-interval (age 4 days) is equal to the given strain at age 4 days. Continuing the process, $V_6 = V_2 + S_6 \cdot C_6$, with S_6 chosen to give the required value of strain at age 8 days, and so on. Stress increments once applied are never removed; but in accordance with the principle of superposition a reduction in stress is represented by the addition of a stress increment of negative sign. The total stress acting at any time is equal to the sum of all S values up to and including that time. The method is adapted to a tabular arrangement which permits rapid computation. In an actual case, the time intervals would be chosen shorter than those used in the example. These time intervals need not be uniform but may be fitted to the variations in the given strain curve. If the problem is one involving two or three dimensions, then corrections should be made for the effect of Poisson's ratio before converting into stress.

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DISCUSSION

MR. HERBERT LEADERMAN[1] (*presented in written form*).—The author is to be congratulated on his very interesting paper, in which the problem of creep is approached fundamentally, and then applied practically, taking into account the complicating factor of aging in concrete.

As he mentioned in his paper, synthetic resins manifest similar creep and recovery phenomena. In general, all organic high-polymeric materials—that is, materials formed of long molecular chains consisting predominantly or entirely of carbon atoms—manifest time effects in their elastic and plastic properties. Such materials include plastics, textile fibers, and rubber, as well as rubber-like materials and materials of biological origin such as muscle. The creep and recovery behavior of some of these materials may be very marked, and for this reason the phenomenon of delayed elasticity may be studied in such materials with extremely simple apparatus.[2] Glass and metals such as silver manifest at room temperature a similar behavior, but in these materials the strains are of the same order of magnitude as in concrete.

About the time of Boltzmann, many investigations were made on the creep, creep recovery, and stress relaxation of glass, silver, and other materials. These investigations were chiefly of academic interest, but had also a practical application. A study of these data[3] shows that in all cases the elastic and delayed elastic behavior obeys the principle of superposition. This suggests, if irreversible deformations are absent, that the superposition principle should be used as a basis for studying the delayed elastic (creep and recovery) behavior of materials.

As a result of comparatively recent developments in the knowledge of the structure of high polymers, it is now possible to give a fairly good picture for the mechanism giving rise to the observed elastic (including delayed elastic) behavior. Three structural types can be found in high polymers. In one case each long molecular chain is assumed to run alternately through amorphous regions, occupied by similar chains randomly arranged, and through crystalline regions where the chains are bound together in a more or less regular three-dimensional arrangement. This structure is represented in the accompanying Fig. 1. Such a material is regenerated cellulose film or filament (viscose rayon). In another type of structure, the regular crystalline regions are absent, but in this case the chains are bound together by chemical cross-bonds. Such materials are thermosetting phenolic and alkyd resins. In the third type of structure, there are neither crystalline regions nor chemical cross-bonds, but merely points of entanglement between the chains, as in the thermoplastic materials.

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[1] Department of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

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We may imagine that relatively weak secondary bonds exist between adjacent segments of chain in the amorphous regions of the material. It is now believed that time effects arise in high polymers, not due to the existence of two *phases*, but due to the presence of two *types of bonds*, namely, primary valence (chemical) bonds in the chains themselves, and the secondary bonds between the chains. In these structures we assume that, between the "fixed points" (that is, crystalline regions, cross-bonds, or entanglement points, respectively) segments of chain are in thermal agitation. As a result, the secondary bond between adjacent segments of chain may be statistically broken and reformed in a slightly different position from time to time. Normally, in a material free from load, the breaking and reformation of secondary bonds occurs in a random manner. If now a small *deformation* is applied to the system, an elastic stress appears, due chiefly to the elastic bending of the chains. This stress corresponds to the "instantaneous" or "acoustic" modulus of elasticity. Under these circumstances the breaking and reformation of secondary bonds are no longer random, but are encouraged to take place in the direction which will relieve the stress in the molecular chains. Hence a gradual relaxation of stress occurs. The equilibrium stress, which is approached asymptotically, corresponds to a relatively small "total" modulus of elasticity.

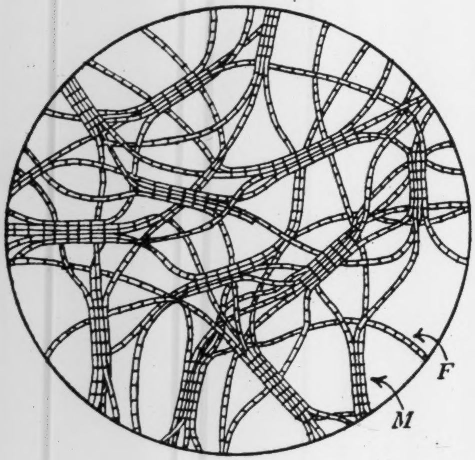

FIG. 1.—Fringe structure of rubber after Herrmann and Gerngross. *F*, fringes; *M*, micelles (crystallites).

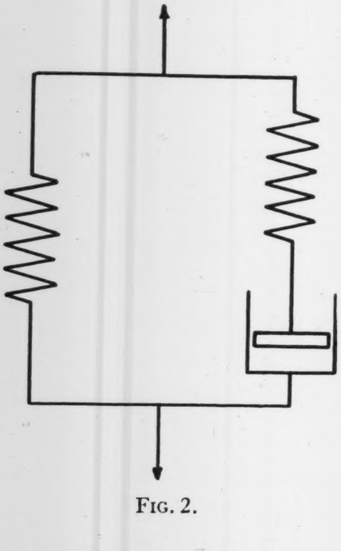

FIG. 2.

If alternatively a constant *load* be applied to the previously unloaded body, there will be an instantaneous elastic deformation, followed by a delayed elastic deformation which ultimately reaches a limiting value. Removal of load results in a (negative) instantaneous deformation, followed by gradual creep recovery to the initial form. The rate at which equilibrium is approached depends upon the strength of the secondary bonds, the temperature, and the lengths of the vibrating segments of chain. In hard plastics equilibrium is approached very slowly, hence these materials have a high apparent modulus of elasticity. In rubber-like materials, such as heated or plasticized thermoplastic resins, equilibrium is approached almost immediately, hence these materials have a low apparent modulus.

It can be shown that the behavior of such structures containing both primary and secondary bonds may be represented most simply by the model shown in the accompanying Fig. 2, in which a spring (with infinite relaxation time) is coupled in parallel with a spring and dashpot (with finite relaxation time). The deformation of this model, as a function of the logarithm of time, due to a load applied at zero time, can be represented in general by a curve such as is shown in the accompanying Fig. 3. The experimentally obtained curves do not correspond however to the simple model of Fig. 2. It appears that we are justified in assuming a *continuous distribution* of relaxation times, following Wiechert.[4] For practical purposes, however, it is satisfactory to assume two discrete relaxation times, as in the present paper. All such models obey the principle of superposition. Hence the chemical structure of plastics is consistent with their elastic behavior.

In conclusion, if there exist in the structure strong primary bonds together with weak secondary bonds, and if the secondary bonds break up and reform as a result of thermal agitation of the elements of the structure, we may expect a delayed elastic behavior following the principle of superposition. This delayed elastic behavior accounts in many cases for phenomena such as elastic hysteresis,[2] damping, and rebound, as well as the softening of thermoplastics and the "brittle point" of rubber-like materials.

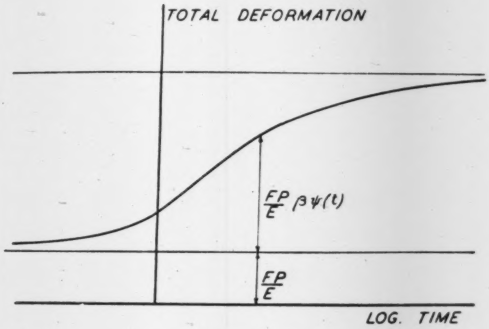

FIG. 3.

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[4] E. Wiechert, "Gesetze der elastischen Nachwirkung für constante Temperatur," Wied. *Annalen der Physik*, Vol. 50, pp. 335, 546 (1893).

STRENGTH OF CONCRETE AS RELATED TO ABRASION OF THE BLAST-FURNACE SLAG USED AS COARSE AGGREGATE

By Fred Hubbard[1] and H. T. Williams[2]

Synopsis

Experience in the field indicates that the abrasion of blast-furnace slag, whether measured by the modified Deval or Los Angeles method, does not predict the strength-producing value of the material when used as concrete aggregate. The characteristics of slag from fifteen sources have been studied together with strength tests of the concrete resulting from their use. The unit weight of the slag does not predict the strength of concrete or the abrasion loss of the slag aggregate. Abrasion tests of slag show a marked similarity between the results of the Los Angeles and Deval methods, although there is a difference in the magnitude of the results. These data confirm field observations that, judging by the compressive and flexural strengths of the resultant concrete, the abrasion tests are not significant in determining the quality of blast-furnace slag aggregate.

In recent years the Los Angeles abrasion test has come into popularity as a specified method of evaluating certain quality characteristics of coarse aggregates. It has been substituted for the Deval abrasion test by many specification-writing groups, chiefly for two reasons: first, only about 15 min. are required for performing the Los Angeles abrasion test, whereas the Deval test requires approximately 5 hr.; second, the Los Angeles method is quite accurate, as the results obtained by various operators working in different laboratories check very closely.

This investigation was undertaken to determine the significance of the Los Angeles abrasion test as a means of evaluating the quality of slag when used as aggregate for concrete. Previous studies[3, 4] have shown that the quality of concrete is not affected by variations in the Deval abrasion of blast-furnace slag used as coarse aggregate. None of these previous investigations, however, included data relative to Los Angeles abrasion, as this test was not made on the aggregates used.

Scope of the Investigation

Samples of blast-furnace slags were selected from 15 commercial sources having a rather wide range in abrasion losses. Determinations were made of the chemical and physical characteristics of the slags and these materials were incorporated in concrete for compressive and flexural strength tests. The same sand and cement were used throughout the series, and the concrete was designed for equal cement content, the amount of mixing water being such as to produce the same consistency in all mixes as represented by an approximately $3\frac{1}{4}$-in. slump. Compressive strength tests were made on 6 by 12-in. concrete cylinders and flexural strength tests on 6 by 6 by 40-in. concrete beams.

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[3] Report of Section on Relation Between the Abrasion Loss on Blast-Furnace Slag and Its Concrete-Making Properties, Appendix VI, Report of Committee C-9 on Concrete and Concrete Aggregates, *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 545 (1930).

[4] Fred Hubbard, "Studies on Relation Between Characteristics of Blast-Furnace Slag and Other Coarse Aggregates and the Properties of Resultant Concretes," Appendix II, Report of Committee C-9 on Concrete and Concrete Aggregates, *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 297 (1936).

MATERIALS

Cement:

Type I portland cement was used, it being selected at one time from a bin of tested cement in sufficient quantity for the entire series of tests.

Chemical analysis per cent:

SiO_2	21.25
Fe_2O_3	3.62
Al_2O_3	5.96
CaO	64.94
MgO	1.15
SO_3	1.55
Loss on ignition	1.13

Physical analysis:

Fineness, passing No. 200 sieve	90 per cent
Surface area (Wagner)	1730 sq. cm. per g.
Initial set	1 hr., 42 min.
Final set	5 hr., 36 min.
Autoclave expansion	0.084 per cent

Tensile strength, psi., of 1:3 sand briquettes:

1 day	169
3 days	315
7 days	399
28 days	489

Sand:

Natural sand having the following characteristics was used as fine aggregate:

Sieve analysis:

Passing No. 4 sieve	99.9	per cent
Passing No. 8 sieve	87.4	per cent
Passing No. 16 sieve	63.0	per cent
Passing No. 30 sieve	36.2	per cent
Passing No. 50 sieve	12.4	per cent
Passing No. 100 sieve	1.0	per cent
Fineness Modulus	3.00	

Physical analysis:

Amount finer than No. 200 sieve, per cent	0.3
Colorimetric test	No. 1
Weight, lb. per cu. ft., dry loose	100.4
Weight, lb. per cu. ft., dry rodded	109.2
Absorption, per cent	0.8
Specific gravity, dry	2.62

Coarse Aggregates:

Blast-furnace slag from 15 sources, representing a total annual production of 4,550,000 tons of commercial slag, was used as coarse aggregate. The slag was oven-dried and separated on square opening sieves into fractional sizes and then recombined for the concrete mixtures in the following proportions:

Passing $1\frac{1}{2}$-in. sieve	100 per cent
Passing 1-in. sieve	62 per cent
Passing $\frac{3}{4}$-in. seive	43 per cent
Passing $\frac{1}{2}$-in. sieve	24 per cent
Passing $\frac{3}{8}$-in. sieve	14 per cent
Passing No. 4 sieve	0 per cent

The chemical and physical characteristics of the slag are shown in Tables I and II.

PROCEDURE

Tests of cement, aggregates, and concrete were made according to the A.S.T.M. methods listed below, except that the specific gravity of the fine aggregate was determined by means of a pycnometer, and the concrete was mixed in a mechanical mixer.

METHODS OF TESTING

Chemical Analysis.—A.S.T.M. Standard Methods of Chemical Analysis of Portland Cement (C 114 – 42).[5]

Unit Weight.—A.S.T.M. Standard Method of Test for Unit Weight of Aggregate (C 29 – 42).[6]

Abrasion.—A.S.T.M. Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 39).[7]

A.S.T.M. Tentative Method of Test for Abrasion of Gravel by Use of the Deval Machine (D 289 – 42 T).[8]

Sieve Analysis.—A.S.T.M. Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 – 39).[9]

Specific Gravity and Absorption:

Coarse Aggregate—A.S.T.M. Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 42).[10]

Fine Aggregate—Bulk specific gravity of the fine aggregate was determined by means of a 1-qt. capacity pycnometer. A 2500-g. sample of saturated sand was surface-dried as prescribed by the A.S.T.M. Standard Method of Test for Specific Gravity and Absorption of Fine Aggregate, (C 128 – 42).[11] One thousand grams of the surface-dried sand were used in the pycnometer for bulk specific gravity determination and 1000 g. of the surface-dried sand were oven-dried and weighed for absorption results.

Voids.—A.S.T.M. Standard Method of Test for Voids in Aggregate for Concrete, (C 30 – 37).[12]

Material Finer than No. 200 Sieve.—A.S.T.M. Standard Method of Test for Amount of Material Finer than No. 200 Sieve in Aggregates, (C 117 – 37).[13]

Colorimetric Test.—A.S.T.M. Standard Method of Test for Organic Impurities in Sands for Concrete, (C 40 – 33).[14]

Yield of Concrete.—A.S.T.M. Standard Method of Test for Yield of Concrete, (C 138 – 39).[15]

Slump of Concrete.—A.S.T.M. Standard Method of Slump Test for Consistency of Portland-Cement Concrete, (C 143 – 39).[16]

Strength of Concrete:

Compressive.—A.S.T.M. Standard Method of Test for Compressive Strength of Concrete, (C 39 – 42),[17] except that the concrete was mixed in a mechanically operated laboratory mixer.

Flexural.—A.S.T.M. Standard Method of Test for Flexural Strength of Concrete (Laboratory Method Using Simple Beam with Third Point Loading) (C 78 – 39),[18] except that the concrete was mixed in a mechanically operated laboratory mixer.

Concrete mixes were designed for a cement factor of 6 sacks per cu. yd. of concrete, with the proportion of coarse aggregate by dry-rodded volume per unit volume of concrete (b/b_0) being 0.71.

TABLE I.—CHEMICAL ANALYSES OF SLAG COARSE AGGREGATE.

Aggregate	SiO_2, per cent	Al_2O_3, per cent	Fe_2O_3, per cent	MnO, per cent	CaO, per cent	MgO, per cent	S, per cent	Total
No. 1	34.78	14.67	0.63	1.62	39.87	6.15	1.29	99.01
No. 2	34.16	13.30	0.50	1.44	43.68	5.01	1.56	99.65
No. 3	37.72	10.15	0.75	2.34	37.38	9.48	1.20	99.02
No. 4	34.88	11.50	0.50	0.99	43.60	6.08	1.57	99.12
No. 5	35.60	13.77	0.63	1.20	41.64	5.16	1.64	99.64
No. 6	34.64	13.77	0.75	1.55	41.02	6.17	1.19	99.09
No. 7	35.00	13.54	0.50	1.31	41.64	6.09	1.49	99.37
No. 8	33.96	14.85	0.63	1.31	43.77	3.33	1.54	99.39
No. 9	37.64	9.88	0.50	1.41	40.92	7.78	1.42	99.55
No. 10	37.60	10.96	0.45	1.60	41.85	6.45	1.38	100.29
No. 11	36.08	11.72	0.50	1.29	42.71	6.48	0.77	99.55
No. 12	36.00	12.48	0.50	1.64	40.40	7.28	1.22	99.52
No. 13	37.28	13.88	0.50	1.28	41.55	3.67	1.41	99.57
No. 14	36.34	15.53	0.37	1.22	42.80	2.11	1.47	99.84
No. 15	44.12	11.53	0.25	1.02	36.94	5.27	0.96	100.09
Average	36.39	12.77	0.53	1.41	41.32	5.77	1.34	99.52

The ingredients were mixed in a mechanical mixer in sufficient quantity to make six 6 by 12-in. concrete cylinders and two 6 by 6 by 40-in. concrete beams from a single batch. The cylinders were capped 4 hr. after fabrication with high-early-strength cement paste, both cylinders and beams remaining in the laboratory at room temperature for 24 hr., after which they were placed in a moist storage room at 70 F., where they remained until tested. Three cylinders and one beam giving three breaks were tested at the age of 7 and 28 days for compressive and flexural strength, respectively.

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[5] 1942 Book of A.S.T.M. Standards, Part II, p. 14.

[6] *Ibid.*, p. 379.

[7] *Ibid.*, p. 359.

[8] *Ibid.*, p. 1238.

[9] *Ibid.*, p. 369.

[10] *Ibid.*, p. 371.

[11] *Ibid.*, p. 373.

[12] *Ibid.*, p. 381.

[13] *Ibid.*, p. 367.

[14] *Ibid.*, p. 366.

[15] *Ibid.*, p. 408.

[16] *Ibid.*, p. 399.

[17] *Ibid.*, p. 391.

[18] *Ibid.*, p. 403.

TABLE II.—UNIT WEIGHT, DEVAL AND LOS ANGELES ABRASION LOSS OF SLAG COARSE AGGREGATE.

Aggregate	Unit Weight, lb. per cu. ft.		Los Angeles Abrasion Loss				Deval Abrasion Loss				Bulk Specific Gravity, Dry	Absorption, 24 hr., per cent	Voids, per cent	
	Loose	Compact	*A*	*B*	Average	Relative Value	Modified *B*	Modified *C*	Average	Relative Value			Loose	Compact
No. 1	63.6	72.0	40.44	38.26	39.35	111	23.66	20.50	22.08	124	2.07	3.9	50.7	44.2
No. 2	63.4	73.2	36.70	34.94	35.82	101	17.10	15.86	16.48	92	2.09	3.5	51.3	43.8
No. 3	64.1	73.2	42.34	40.14	41.24	116	24.96	20.70	22.83	128	2.08	3.7	50.5	43.6
No. 4	66.4	76.4	32.58	30.94	31.76	90	17.10	15.10	16.10	90	2.18	3.2	51.1	43.8
No. 5	69.4	79.2	31.70	30.90	31.30	88	15.66	15.76	15.71	88	2.22	3.3	49.9	42.8
No. 6	69.6	79.4	36.74	35.10	35.92	101	18.16	15.50	16.83	94	2.21	2.9	49.5	42.4
No. 7	71.4	81.2	34.74	32.10	33.42	94	16.16	15.10	15.63	87	2.29	2.4	50.0	43.1
No. 8	72.0	82.0	31.18	30.04	30.61	86	16.26	15.06	15.66	88	2.28	2.9	49.4	42.3
No. 9	72.4	82.2	41.30	38.84	40.07	113	23.46	21.76	22.61	127	2.23	2.8	47.9	40.9
No. 10	73.2	82.4	41.60	40.14	40.87	115	20.70	18.34	19.52	109	2.32	2.0	49.3	43.0
No. 11	72.8	82.8	32.24	31.50	31.87	90	17.06	15.30	16.18	91	2.30	2.3	49.2	42.3
No. 12	74.2	84.0	34.56	34.04	34.30	97	16.50	15.30	15.90	89	2.31	2.2	48.5	41.7
No. 13	75.0	84.2	34.74	34.98	34.86	99	17.76	18.20	17.95	100	2.29	2.2	47.5	41.0
No. 14	74.8	84.4	36.84	36.58	36.71	104	18.20	16.20	17.20	96	2.39	2.0	49.8	43.4
No. 15	88.0	98.6	33.40	34.20	33.82	95	16.54	18.26	17.40	97	2.67	0.6	47.1	40.7
Average	71.4	81.0	36.07	34.84	35.46	100	18.61	17.12	17.87	100	2.26	2.7	49.3	42.6

TABLE III.—PROPORTIONS OF MATERIALS AND PHYSICAL TESTS OF FRESH CONCRETE.

Aggregate	Materials per cubic yard of Concrete, Computed from the Unit Weight of the Fresh Concrete					Slump of Concrete, in.	Weight of Concrete, lb. per cu. ft.
	Sacks of Cement by Weight of Concrete	Dry Sand, lb.	Dry Slag, lb.	Net Water, gal. per sack	Net Water, cu. ft. per sack		
No. 1	5.93	1382	1363	6.18	0.83	3¼	135.4
No. 2	5.95	1377	1392	6.08	0.81	3¼	136.1
No. 3	5.96	1370	1394	6.19	0.83	3½	136.3
No. 4	5.95	1374	1452	6.21	0.83	3¼	138.4
No. 5	5.93	1340	1501	6.20	0.83	3½	138.9
No. 6	5.97	1327	1514	6.13	0.82	3½	138.8
No. 7	5.92	1346	1535	6.20	0.83	3¼	140.0
No. 8	5.96	1331	1561	5.97	0.80	3½	140.4
No. 9	6.00	1295	1576	5.91	0.79	3½	139.7
No. 10	5.98	1357	1574	6.01	0.80	3¼	141.7
No. 11	5.96	1329	1575	6.11	0.82	3½	140.9
No. 12	5.99	1317	1605	6.02	0.80	3¾	141.5
No. 13	6.00	1292	1614	5.93	0.79	3½	141.1
No. 14	5.91	1353	1594	5.99	0.80	3¼	141.6
No. 15	5.96	1284	1879	6.03	0.81	3¼	149.7
Average	5.96	1338	1541	6.07	0.81	3⅜	140.0

DISCUSSION OF TEST DATA

Table I shows the chemical analyses of the slags. Sample No. 15 has an unusually high silica (SiO_2) content and has the highest weight per cubic foot of the slags tested in this series. In general, the more dense or heavy slags are those in which the silica content is relatively high.

TABLE IV.—COMPRESSIVE AND FLEXURAL STRENGTHS OF CONCRETE.

Each age result is the average of three tests.

Aggregate	Compressive Strength, psi.				Flexural Strength, psi.			
	7 days	28 days	Avg.	Relative Value	7 days	28 days	Avg.	Relative Value
No. 1	4658	6366	5512	107	787	838	813	102
No. 2	4594	6043	5319	103	786	917	852	107
No. 3	4532	6310	5421	105	812	773	793	100
No. 4	4080	5334	4707	91	763	789	776	98
No. 5	4267	5895	5081	98	769	855	812	102
No. 6	4286	5858	5072	98	750	855	803	101
No. 7	4641	6150	5376	105	795	757	776	98
No. 8	4274	5885	5080	98	779	857	818	103
No. 9	4715	5835	5275	102	772	788	780	98
No. 10	4342	5952	5147	100	801	817	809	102
No. 11	4493	5966	5230	101	750	823	787	99
No. 12	4571	6055	5313	103	735	735	735	93
No. 13	4028	5116	4572	89	746	827	787	99
No. 14	4456	6092	5274	102	831	821	826	104
No. 15	4418	5547	4982	97	682	807	745	94
Average	4424	5893	5159	100	771	817	794	100

Table II shows the unit weights and Los Angeles and Deval abrasion losses of the slags. These data are compared graphically in Figs. 1 and 2. It also gives the specific gravity, absorption, and voids of the aggregates.

Table III shows the concrete proportions expressed in quantities of the various ingredients required per cubic yard of concrete. These proportions are based upon the yield computed from the unit weight of the fresh concrete. The table

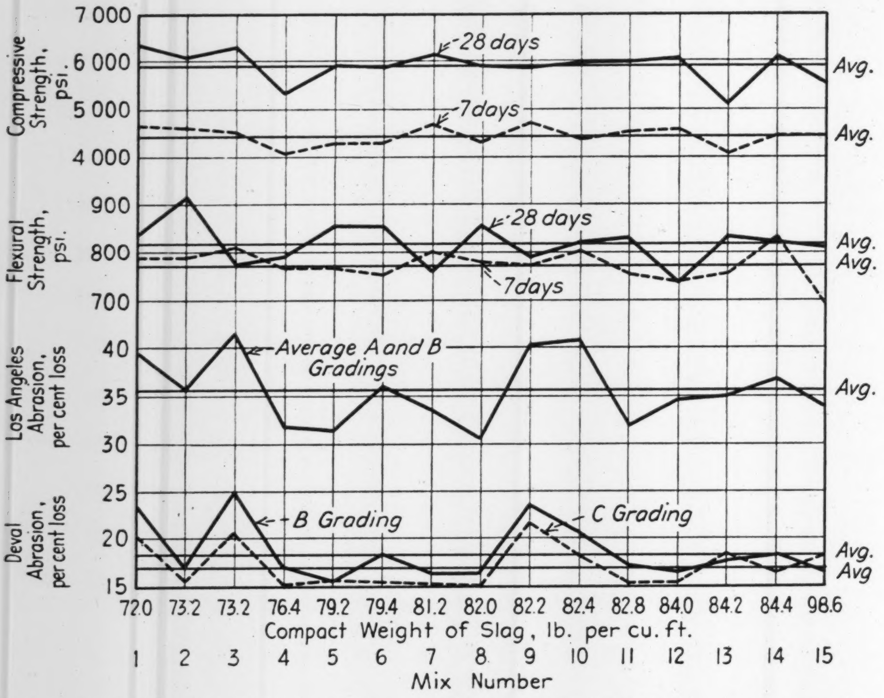

FIG. 1.—Relation of the Unit Weight of Slag to Abrasion Loss and the Strength of Concrete.

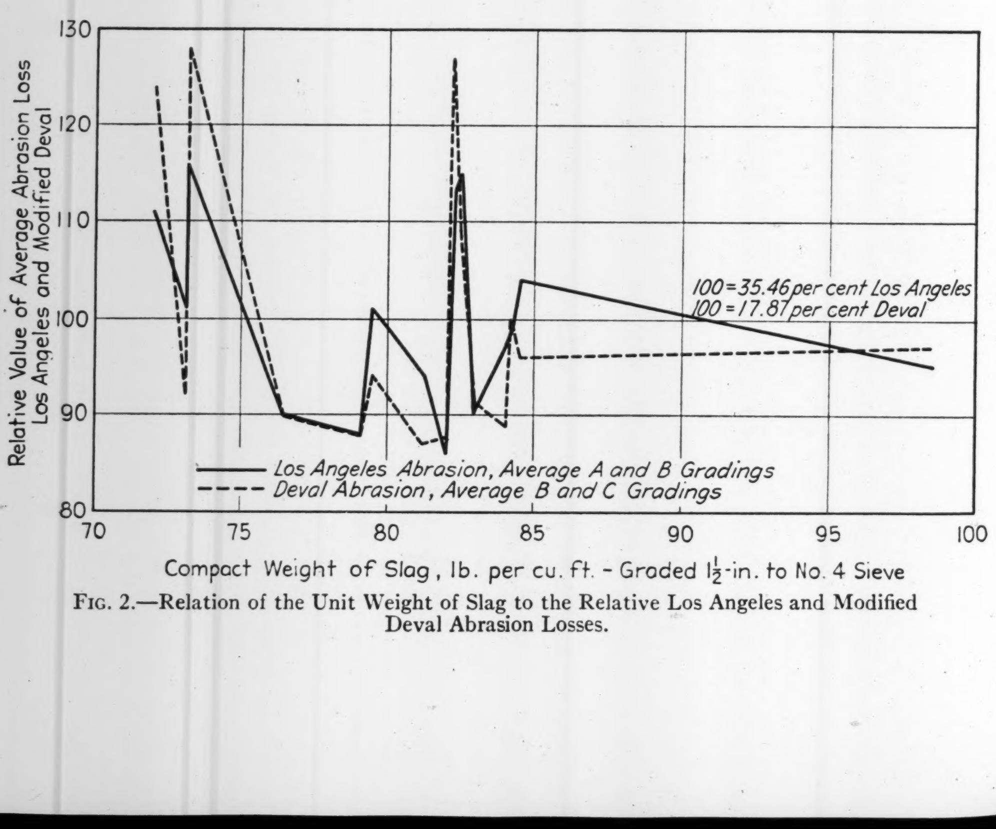

FIG. 2.—Relation of the Unit Weight of Slag to the Relative Los Angeles and Modified Deval Abrasion Losses.

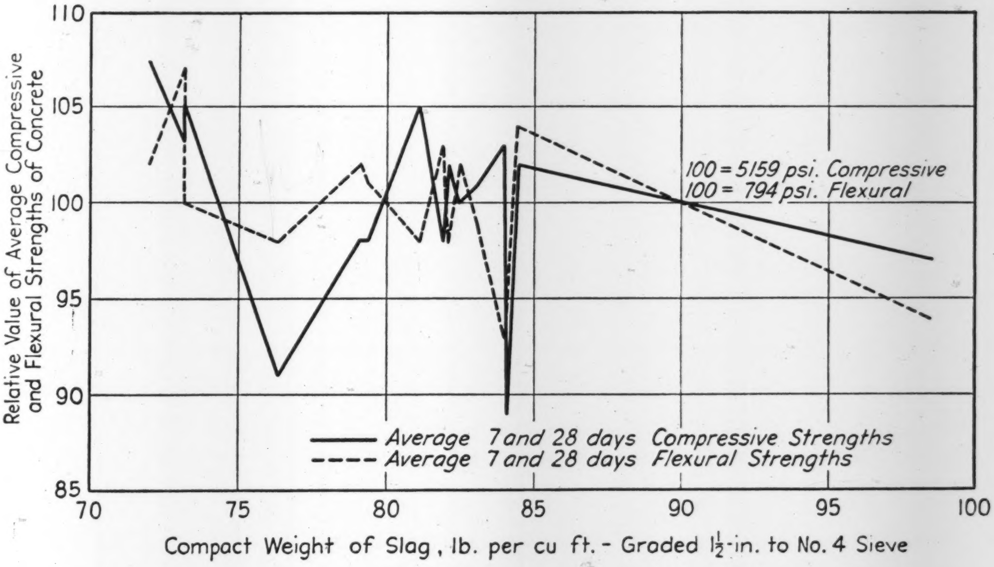

FIG. 3.—Relation of the Unit Weight of Slag to the Relative Value of Compressive and Flexural Strengths of Concrete.

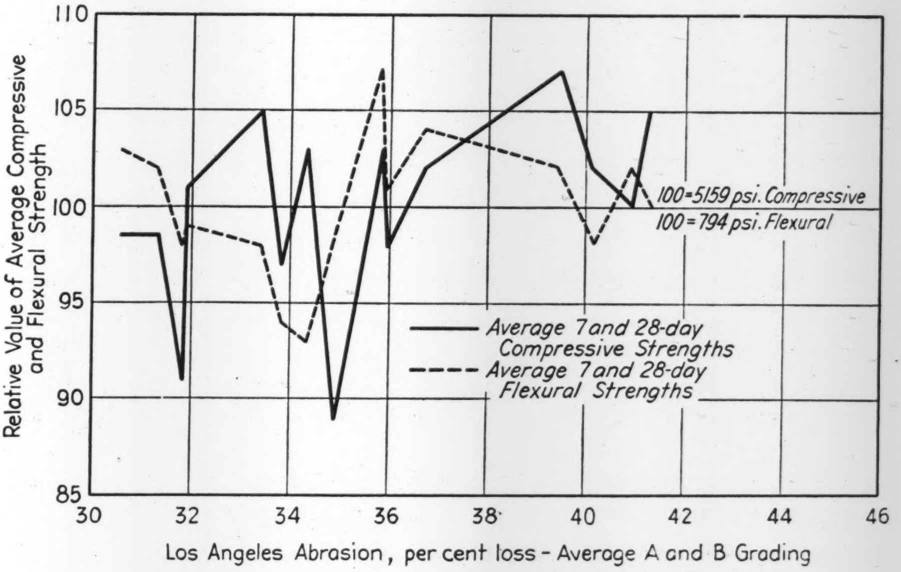

FIG. 4.—Relation of the Los Angeles Abrasion of Slag to Relative Value of Compressive and Flexural Strengths of Concrete.

also shows slump and unit weight of the concrete.

It will be noted that while the mixes were designed on the basis of specific gravity for 6 sacks of cement per cu. yd., yet in most instances the cement factor, computed from the unit weight of the fresh concrete, was slightly less than this, averaging 5.96 sacks per cu. yd.

Table IV shows the compressive and flexural strength of the concrete at 7 and 28 days with their relative values based on the average of all strengths as being 100 per cent. These data are compared graphically with the unit weight and abrasions of the slag aggregates in Figs. 1, 3, and 4.

Figure 1 shows the relation between the unit weight of slag graded $1\frac{1}{2}$-in. to No. 4 sieve, the abrasion losses of the slag, and the strength of the resulting concrete. The range in unit weight of the slag by dry-rodded volume is from 72.0 to 98.6 lb. per cu. ft. There is no coincidence between the unit weight of the slag and the abrasion loss or strength of concrete.

Figure 2 shows the relation of unit weight of slag to the relative Los Angeles and modified Deval abrasion losses. In the scope of this study there is no direct relationship between the unit weight of slag and either the Los Angeles or Deval abrasion losses. These data do show a remarkable similarity between the relative values of the Los Angeles and modified Deval abrasion results.

Figure 3 shows the relation of the weight of slag to relative values of the average 7 and 28 day compressive and flexural strengths of the resultant concrete. Variations in the weight of slag used as coarse aggregate from 72.0 to 98.6 lb. per cu. ft. does not affect either the compressive or flexural strength of the concrete. The strength of concrete resulting from the use of slag from the various sources is very uniform. Only three materials produced concrete which varied more than 5 per cent from the average compressive strength and in only two instances did flexural strength vary more than 5 per cent from the average.

Figure 4 shows a comparison between the Los Angeles abrasion and the relative values of the average 7 and 28 day compressive and flexural strength of the concrete. The range in the average Los Angeles abrasion losses of the slag aggregates is 30.61 to 41.24 per cent. Differences in the Los Angeles abrasion of the slags studied do not show any effect on the strength of concrete.

CONCLUSIONS

1. There is no direct relationship between the Los Angeles abrasion results of blast-furnace slag and the compressive or flexural strength of concrete in which it is used as coarse aggregate. See Tables II and IV, and Figs. 1 and 4.

2. These results confirm the conclusions of previous investigations that no relationship exists between the modified Deval abrasion loss of blast-furnace slag coarse aggregate and the strength of concrete. See Tables II and IV, and Fig. 1.

3. There is a definite similarity between the abrasion losses of blast-furnace slag when measured by the Los Angeles and the modified Deval methods. The percentage of loss by the Los Angeles method is approximately twice the modified Deval abrasion loss. See Table II and Fig. 2.

4. The unit weight of slag does not predict the magnitude of the Los Angeles or Deval abrasion results or the strength of the resultant concrete. See Tables II and IV, and Figs. 1, 2 and 3.

5. Judged by the compressive and flexural strengths of the resulting concrete, the Los Angeles and Deval abrasion tests are not significant in determining the quality of blast-furnace slag for use as concrete aggregate.

DISCUSSION

Mr. J. E. Boyd[1] (*presented in written form*).—The Los Angeles abrasion test has been widely adopted as a specified method of evaluating coarse aggregates only after long and careful investigation, embracing not fifteen sources of supply of one particular kind of aggregate but hundreds of sources including all types of coarse aggregate.

In the report of the Highway Research Board Committee on Correlation of Research in Mineral Aggregates,[2] we find that aggregates ranging in Los Angeles abrasion from 11 per cent to 74 per cent, were studied. These aggregates, of course, not only varied in percentage of wear but in surface texture, in shape, and in composition. However, a very definite relation between the loss in the Los Angeles test and the strength of concrete is found. Graphs included in this report show that the flexural strength of 1.5 cement factor concrete increases at the rate of about 50 psi. for each 10 per cent reduction in Los Angeles abrasion loss.

The range in abrasion loss of slags examined by the authors is not sufficiently wide to establish the trend. By all previous data, a variation of 50 psi. modulus of rupture is all that could be expected between the slags of the lowest and highest Los Angeles losses considered; and by the figures of the authors, the factor of error in the flexural strength test itself must exceed the difference in strength that might otherwise be attributed to the small variations in abrasion.

Perhaps there are no blast-furnace slags with Los Angeles losses below 30 per cent or above 40 per cent (gradation B). If not, would not this paper have been more appropriately entitled: The Remarkable Uniformity of Blast Furnace Slag? The chemical analyses presented are remarkably uniform. The surface texture of slag is no doubt very uniform, and so is the particle shape. I imagine that the true specific gravity of slag of such uniform chemical composition is also quite uniform. The bulk specific gravity is not uniform, indicating that some slags retain more gases in occluded voids than do others. And here we may note that the Los Angeles loss for gradation A is consistently higher than the loss for gradation B. This I believe is the reverse of results obtained with gravel and stone. Is it not true that the bulk specific gravity of slag increases with reduction in the size of the particle? Are not the larger pieces of slag lighter in unit weight than the smaller pieces? Does not that fact explain the higher loss for gradation A than for gradation B?

The absorption is not uniform. It increases with porosity or as the unit weight decreases. And are the strength results so very uniform? The authors have averaged 7- and 28-day results of each sample and compared this average with the averaged average of the whole 15 samples showing a maximum deviation of 11 per cent in compressive strength and a maximum deviation of 7 per cent in flexural strength. Comparisons of individual strength results show maximum variations of 17 per cent for

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[1] Sales Engineer, The Weston & Brooker Co., Atlanta, Ga.

[2] *Proceedings*, Seventeenth Annual Meeting, Highway Research Board, Vol. 17, p. 350 (1937).

7-day compressive strength, 24.4 per cent for 28-day compressive strength, 22 per cent for 7-day flexural strength, and 24.8 per cent for 28-day flexural strength.

I am much impressed by the very high strengths reported, especially the high 7-day strengths. Aggregates Nos. 3, 7, and 14 produce 7-day flexural strengths higher than their 28-day flexural strengths, and aggregate No. 12 shows no gain in 28-day over 7-day flexural strength. The reported average 28-day flexural strength is only 6 per cent higher than the 7-day strength, whereas the 28-day strength of the cement used increased 23 per cent above the 7-day strength.

Is it not significant that the exceptionally heavy slag, No. 15, produces the lowest 7-day flexural strength? The growth in strength is slower. Will not the date of declining strength be longer postponed?

I am impressed also by the similarity of chemical analyses of these slags to that of portland cement itself. Is it not likely that there is chemical action between the slag and the cement which produces accelerated hardening and rapid development of ultimate strength? Portland cement concrete, of the proportions used by the authors, consists of about 10 per cent portland cement by absolute volume and the coarse aggregate comprises about 50 per cent of the absolute volume. The specifications governing portland cement limit magnesia to 3 per cent, which means magnesia in concrete contributed by portland cement could not exceed 0.3 per cent. The authors show that blast-furnace slag will contain from 2.11 to 9.48 per cent magnesia or an average of about 6 per cent. Slag concrete then would carry about 3 per cent magnesia, or 10 times the amount permitted in concrete through portland cement. Is the magnesia in slag chemically different from magnesia in cement clinker? The particle size of slag, as coarse aggregate, is massive as compared with the cement particle. Accordingly the rate of chemical change within the slag particle must be comparatively very slow but does particle size serve to prevent interaction between the water-soaked surfaces of slag and cement? And does not this surface contact accelerate the crystallization of cement? What force is responsible for higher flexural strengths at 7 days than at 28 days, as in the case of concretes made of slags Nos. 3, 7, and 14?

MESSRS. FRED HUBBARD[3] AND H. T. WILLIAMS[4] (*authors' closure, by letter*).—Mr. Boyd has raised some very interesting questions relative to the value of the Los Angeles abrasion test as applied to slag. The Highway Research Board report to which he refers does not indicate that any tests were made on slag concrete. With one exception all slags tested for abrasion gave satisfactory service records, the exception being a material weighing only 69.5 lb. per cu. ft. Our report does not question the value of the Los Angeles abrasion test as a means of evaluating concrete aggregate other than blast-furnace slag.

The following are the results of 74 Los Angeles abrasion tests of slag from 32 sources representing approximately 90 per cent of the commercial slag produced in the United States:

LOS ANGELES ABRASION TESTS LOSS IN PER CENT OF SLAG FROM 32 SOURCES (74 TESTS).

	Minimum	Maximum	Average
A grading	23.10	47.38	34.37
B grading	22.94	45.70	35.14
Average A and B grading	23.02	45.41	34.77

	Number of Tests	Percentage of Total
Samples with less than 25.00 per cent abrasion	3	4
Samples with less than 30.00 per cent abrasion	12	16
Samples with less than 35.00 per cent abrasion	41	55
Samples with less than 40.00 per cent abrasion	63	85
Samples with less than 45.41 per cent abrasion	74	100

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[3] Director of Research, National Slag Assn., Youngstown, Ohio.

[4] Materials Engineer, Duquesne Slag Products Co., Youngstown, Ohio.

Sixty-nine per cent of all samples tested gave abrasion losses between 30 and 40 per cent. It is true that, generally speaking, the larger pieces of slag are slightly lower in bulk specific gravity than the smaller pieces for the reason that the larger pieces come from those portions of the pits in which the molten slag solidifies in comparatively thick layers thus trapping more of the escaping bubbles of gas or steam than is the case in the thinner layers from which the greater portion of the smaller sizes comes. There is, however, practically no difference between the abrasion losses from the A and B gradings. In the tests referred to above the average abrasion loss of the smaller material (B grading) is slightly greater than the A grading although the difference is too small to be significant.

Averaging concrete strengths developed at different ages to determine the relative values of different materials is a usual and logical practice. The strengths developed at the different ages are clearly shown both in Table V and also in Fig. 1, from which direct comparison can be made for each age. While the strengths of the concrete are high, yet they are no higher than those obtained in the field with slag aggregate as indicated by the results reported by Highway Departments and others. For illustration, Federal Aid Concrete Paving Project No. 552 I and 552 B, constructed in 1940 on Traffic Route U. S. 422 in Mahoning County, Ohio, used slag aggregate with six sacks of cement per cubic yard of concrete, the strengths of which, as reported by the Highway Department, were as follows:

Compressive Strengths:

7 days, average of 49 cylinders 3949 psi.
28 days, average of 56 cylinders 5559 psi.

Flexural Strengths:

7 days, average of 112 beam breaks 768 psi.
(No 28-day tests made)

Federal Aid Project No. 606-g (1) constructed in 1941 on Traffic Route U. S 35 in Jackson County, Ohio, used slag aggregate with six sacks of cement per cubic yard, which developed the following strengths:

Compressive Strengths:

7 days, average of 18 cylinders 4630 psi.
28 days, average of 20 cylinders 5880 psi.

Flexural Strengths:

7 days, average of 38 beam breaks 815 psi.
(No 28-day tests made)

Judging from the above field results, the strengths given in the report are not unusual for laboratory fabricated concrete and can be equalled by well-controlled field concrete.

We do not believe that there is any significance to the 7-day flexural strength resulting from the use of slag No. 15. The 28-day strength of this material is approximately equal to the average of all fifteen materials tested. In the presence of moisture the strength of concrete should continue to increase indefinitely with age. If allowed to dry out the concrete will decrease in strength somewhat but concrete made from the lighter slags weighing 70 to 80 lb. per cu. ft. will behave no differently in this respect than concrete made from the heavier slags, crushed stone or gravel. This fact is illustrated by a report by one of the authors entitled "Studies on Relations Between Characteristics of Blast-Furnace Slag and Other Coarse Aggregates and the Properties of Resultant Concretes," presented before the Society in 1936, with particular reference to Fig. 4, and Table XII.[5]

The latest A.S.T.M. Standard Specifications for Portland Cement (C 150 – 42)[6] limit the magnesium oxide (MgO) content to 5 per cent for types I, II,

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[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, pp. 312 and 313 (1936).

[6] 1942 Book of A.S.T.M. Standards, Part II, p. 1.

III, and IV cement and 4 per cent for type V cement. The magnesia content of slag in combination as Melilite of 2.11 to 9.48 may be considered as an average range for blast-furnace slag. It is not unusual for natural aggregates to have a magnesia content as high as 20 per cent existing in some combined form.

A report by Brownmiller[7] published by the American Concrete Institute states "Delayed expansion of cement is often attributed to the hydration of the MgO to $Mg(OH)_2$ which reaction is accompanied by an increase in volume setting up stresses in the cement."

A report by White and Kemp[8] presented before the Society in 1942 concludes, "The autoclave test evidently detects this free magnesia which had not become hydrated even after immersion in water for 40 yr. Combined magnesia does not cause expansion in either water at room temperature or in the autoclave test. The autoclave test has not caused large expansion in any of the bars except those known to contain free magnesia." Blast-furnace slag does not contain free magnesia. Autoclave tests of blended cements in which as much as 50 per cent of blast-furnace slag is ground in with portland cement clinker show unusually low expansion.

We have no explanation for the fact that in three cases the 28-day flexural strengths are lower than those developed at 7 days other than experimental error. We do know that the flexural strength of slag increases with age the same as the compressive strength when the concrete is subject to moist-curing conditions.

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[7] L. T. Brownmiller, "The Microscopic Structure of Hydrated Portland Cement," *Journal*, Am. Concrete Inst., January, 1943.

[8] Alfred H. White and Harold S. Kemp, "Long-Time Volume Changes of Portland Cement Bars," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 727 (1942).

TESTS OF THE FIRE RESISTANCE AND THERMAL PROPERTIES OF SOLID CONCRETE SLABS AND THEIR SIGNIFICANCE[1]

By Carl A. Menzel[2]

Synopsis

This paper deals mainly with the performance of 16 solid concrete slabs 5⅓ ft. wide by 6 ft. high when exposed on one face to standard fire temperatures. These slabs, cast horizontally of plastic concrete, were tested vertically and simulated solid wall specimens 4, 6, and 8 in. thick. The slabs carried loads (as walls) of 400 or 500 psi. during exposure to fire. After cooling to room temperature, each slab was subjected to a second fire-endurance test. Load-carrying ability as a wall after two exposures to fire was determined.

The tests developed significant information on the thermal properties (heat transmission, expansion, etc.) of solid concrete made with several widely different aggregates: (1) highly siliceous sand and gravel; (2) calcareous sand and gravel; (3) fine and coarse crushed Haydite; (4) mixtures of equal volumes of (1) and (3). Information was also obtained on the effect of variations in the size of the highly siliceous aggregate and on the effect of duration of air drying of the slabs before test.

Relationships are established between slab thickness (or weight per square foot of slab) and the fire-endurance period, for each type of aggregate. Other relationships give the approximate time required to attain a critical temperature (1000 F.) in the reinforcing steel of flexural members at various distances from the exposed face of the slab. The significance, with respect to heat transfer in reinforced concrete floor slabs, of the relationships presented are discussed in the light of data from various sources on the general effect of elevated temperatures on the yield strength and modulus of elasticity of carbon steel.

The various relationships obtained are useful as indicative of the fire resistance that plain and reinforced concrete assemblies can develop, assuming that it is not limited by the degree to which the full-size assembly is stable and maintains its integrity under combined fire exposure and loading or restraint. Their application is illustrated by several comparisons between the estimated and actual performance of typical ribbed-slab assemblies.

Scope, Procedure, and Data of Tests

Introduction:

The effective use of concrete in fire-resistive construction depends in large measure on definite knowledge of its thermal properties and behavior during exposure under controlled fire test conditions. To supply such knowledge two investigations were carried out in the Research Laboratory of the Portland Cement Assn. The first and most comprehensive of these investigations comprised tests of the fire resistance and strength of 215 walls of precast hollow concrete masonry units. These tests brought out the relative influence of various factors of composition, form, and assembly of the units on wall performance, and established the effect of each factor through a wide range of variation in that factor.

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[1] Prepared under the auspices of Committee C-5 on Fire Tests of Materials and Construction of the American Society for Testing Materials.

[2] Associate Engineer, Research Laboratory, Portland Cement Assn., Chicago, Ill.

One of the most useful developments of this research was that in tests of 4, 8, and 12-in. walls, constructed with units of different design but with a single unit through the wall thickness, a remarkably consistent relationship was found between the fire-endurance period (time to attain 250 F. rise on unexposed face) and the weight per square foot of wall. For a given type of concrete there was a marked increase in the fire-endurance period with increase in weight. In general, an increase of 50 per cent in weight doubled the fire endurance period. The relationship appears to be fundamental and to hold regardless of the manner in which the concrete is distributed in the different designs of units. The simple relationships that were developed with six widely different aggregates provided a rational basis for choice of unit to give any desired performance as to fire endurance. The results were reported in two papers (**15**),[3] (**16**), and in a final report (**17**) covering all phases of the research.

Fairly complete information has, therefore, been available on the thermal properties of the different types of concrete found in precast units made by tamping or vibrating dry, harsh mixtures. As a group, these concretes are relatively lean in cement content, have a porous internal structure, and are made with aggregates graded up to a maximum size of $\frac{3}{8}$ or $\frac{1}{2}$ in. Although the concrete mixtures are made as wet as practicable, they are inherently dry in comparison with plastic concrete because the freshly compacted units must have sufficient rigidity to permit removal from the mold to the curing racks immediately after molding.

The present paper presents and discusses the significance of the results obtained in the second investigation which concerned solid slabs made with the plastic concrete commonly used in reinforced concrete construction.

Purpose and Scope:

The chief purpose of the second investigation was to obtain information on the thermal properties, load-carrying ability (as a wall), and behavior with respect to thermal effects of the type of concrete used in plain and reinforced concrete members (particularly in solid bearing walls and floor slabs) of modern fire-resistive construction. Such concretes are cast in place and must be sufficiently plastic to permit effective placing around reinforcing steel, structural steel shapes, or in deep narrow forms with or without steel. Compared with the concrete generally used in precast masonry units, this plastic concrete is richer in cement content, has a denser structure, and is made from a wetter mixture with aggregates graded up to $\frac{3}{4}$, 1, or $1\frac{1}{2}$ in. maximum size instead of to $\frac{3}{8}$ or $\frac{1}{2}$-in. maximum.

In the light of the knowledge available regarding the effect of various factors influencing the properties of dry-tamped concrete, it was possible to obtain much of the information desired on plastic concrete from a series of tests involving 16 solid concrete slabs and 2 rib-type slab specimens. In the 16 solid slabs, thickness of slab, type of aggregate, period of air drying, and size of aggregate were the major variables with cement content, consistency, and method of placing constant. The rib-type specimens brought out the effect of plaster and served to illustrate how the relationships developed from the tests on simple solid slabs may be applied to more complex and composite assemblies.

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[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1146.

General Description of Tests:

The 16 solid slabs were cast horizontally but were tested vertically as load-bearing wall specimens. They were $5\frac{1}{3}$ ft. wide and 6 ft. high and 4, 6, and 8 in. thick and carried loads of 400 or 500 psi. during exposure to fire. The fire endurance and load tests were of the same nature as those employed in the first investigation on walls of hollow concrete masonry units except that after cooling to room temperature, each slab was subjected to a second fire endurance test. The load-carrying ability after two exposures to fire was then determined by applying loads up to the capacity of the testing equipment. Tests on the two rib-type specimens are described later in this paper.

Data on the thermal properties of the concrete were obtained from the rise in temperature indicated by thermocouples embedded at various distances from the face of the slab exposed to the fire as well as by the usual thermocouples and thermometers in contact with the unexposed face of the slab under standard pads. The data on the temperature rise in the interior of the slab provided desired information on (1) the approximate time to attain critical temperatures in reinforcing steel at various distances from the exposed face and (2) the approximate thickness of concrete required to protect reinforcing steel from critical temperatures during various periods of exposure to standard fire temperatures. The relations obtained in this manner on solid slabs provide data of practical significance in the design of reinforced concrete floor slabs.

Testing Technique and Equipment:

The tests were conducted to follow the general procedure described by A.S.T.M. Standard Methods of Fire Tests of Building Construction and Materials (C 19 – 41).[4] The equipment for fire and load tests was the same as used in the first investigation on walls of concrete masonry units.[5] Lack of head and side room made it necessary to limit the capacity of the equipment to specimens not more than $5\frac{1}{2}$ ft. wide and $6\frac{1}{4}$ ft. high. The test slabs used in this investigation were $5\frac{1}{3}$ ft. wide and 6 ft. high with a total area of 32 sq. ft. instead of the 100 sq. ft. total area and 9 ft. minimum dimension specified for standard tests of load-bearing walls. However, to insure obtaining dependable information on the relative influence of various factors studied, a testing technique was adopted which was more exacting then standard requirements in the following respects:

1. Temperatures on unexposed face were indicated by thermocouples of No. 20 B. & S. gage wire which were more sensitive to temperature changes than either the thermocouples of No. 18 B. & S. gage wire or the thermometers permitted by the standard.

2. Temperatures on unexposed face were measured at nine stations over an area of 32 sq. ft. instead of at five stations over an area of 100 sq. ft. as permitted by the standard. Thus there was one temperature station for every 3.5 sq. ft. of unexposed area instead of for every 20 sq. ft.

3. The temperature sensitive point of each furnace thermocouple was placed

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[4] 1942 Book of A.S.T.M. Standards, Part II, p. 196.

At the time of the tests (January 19, 1931 to August 31, 1932) these methods were a tentative standard of the American Society for Testing Materials, C 19 – 26 T. They were adopted in May, 1926, as a standard of the National Fire Protection Assn., and by the Fire Council of Underwriters' Laboratories, and in 1933 as a standard of the American Society for Testing Materials, C 19 – 33. This A.S.T.M. standard, as revised in 1941, became A.S.A. Standard A2.1 in 1942.

[5] The equipment has been fully described in pages 609 to 612 of reference (15) and in pages 3 to 7 of reference (17).

3 in. from the exposed face of the specimen instead of the permissible 6 in.

4. Furnace temperatures were indicated by seven thermocouples distributed over an area of 32 sq. ft. instead of by five thermocouples distributed over an area of 100 sq. ft. Hence there was one thermocouple for every 4.6 sq. ft. of exposed face instead of for every 20 sq. ft.

5. More precise furnace temperature control was attained. Adherence to area under standard furnace control curve was within 1 per cent during the first hour, and within 0.5 per cent for longer periods, instead of the 10 per cent for the first hour, the 7.5 per cent for tests up to 2 hr., and the 5 per cent for tests exceeding 2 hr. permitted by the standard.

6. Test furnace was located in a comparatively small room in which all windows and doors were kept closed during the tests to minimize cooling effect of drafts.

7. Elimination so far as practicable of effect of moisture in test specimens at time of exposure.

The effort made to eliminate the effect of moisture on the thermal properties of the concrete was an important feature of the tests on solid slabs. In order to accomplish this, 14 of the 16 solid slabs were seasoned for 12 to 14 months in freely circulating dry air in the laboratory. Therefore they approached as nearly as practicable the moisture content of plastic concrete exposed to the temperature and humidity of a heated building. For comparison, the other two solid slabs were seasoned for approximately 1 month before testing. Data on the approximate loss of moisture from the slabs during the seasoning period were obtained from the changes in weight of pilot slab specimens 1 ft. square and of the same thickness as the large slabs. The edges of these pilot specimens were coated with paraffin to confine the moisture loss to the two opposite faces. With the precautions taken it is believed that these small specimens served as fairly reliable indicators of the change in the moisture content of the concrete in the large slabs during seasoning. Readings of the changes in weight of these pilot specimens were continued for 12 yr.[6] The record indicates that when the three thickest slabs (8-in.) were exposed to fire at 13 to 14 months, they had lost from 80 to 90 per cent of the total moisture they might have lost during 12 yr. of service in a heated building. Further evidence regarding the effect of moisture was obtained when the slabs were subjected to the second exposure to fire after all of the free water and some of the water combined with the cement had been removed during the first exposure to fire.

The various steps in the routine test procedure were as follows:

(*a*) *Concrete.*—All slabs were made with concrete of plastic consistency placed by hand rodding or spading and having a cement content of 5.2 sacks per cu. yd., and a slump of 5 to 6 in. For further details, see Table II.

(*b*) *Curing and Seasoning.*—The slabs were cast horizontally in steel molds. After moist curing in the mold for 5 days they were removed and stored in a vertical position in the air of the laboratory for 12 to 14 months before exposure to the fire endurance test.[7] To facilitate safe handling at 5-day age each slab was provided with five ½-in. round reinforcing rods placed 1 in. and 1½ in. from the bottom of the mold as shown in Fig. 1.

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[6] Fire tests on the 16 solid slabs were made between January, 1931, and August, 1932, in conjunction with some of the later tests on walls of hollow concrete masonry units of the first investigation. The preparation of this paper was postponed until certain trends of weight changes in some of the small pilot specimens had been well established. These will be referred to later.

[7] In the case of the 2 slabs used for tests Nos. 126 and 127 the moist curing period was 10 days instead of the usual 5 days. These 2 slabs were exposed to fire after storage in air for nominally one month (27 and 32 days) instead of 12 to 14 months for the 14 other slabs tested.

(*c*) *Measurement of Temperatures Within Slab.*—In casting the slabs iron-constantan thermocouples were embedded at the following distances from the bottom of the mold: 1 and 2 in. for 4-in. slabs; 1, 2, and 4 in. for 6-in. slabs, and 1, 2, 4, and 6 in. for 8-in. slabs. The thermocouples 1 in. from the bottom were wired to the lower (fire) side of the $\frac{1}{2}$-in. round reinforcing rods. Since the bottom of the slab as cast was the face exposed to fire, the various couples indicated temperatures within the slab at 1, 2, 4, and 6 in. from the exposed face.

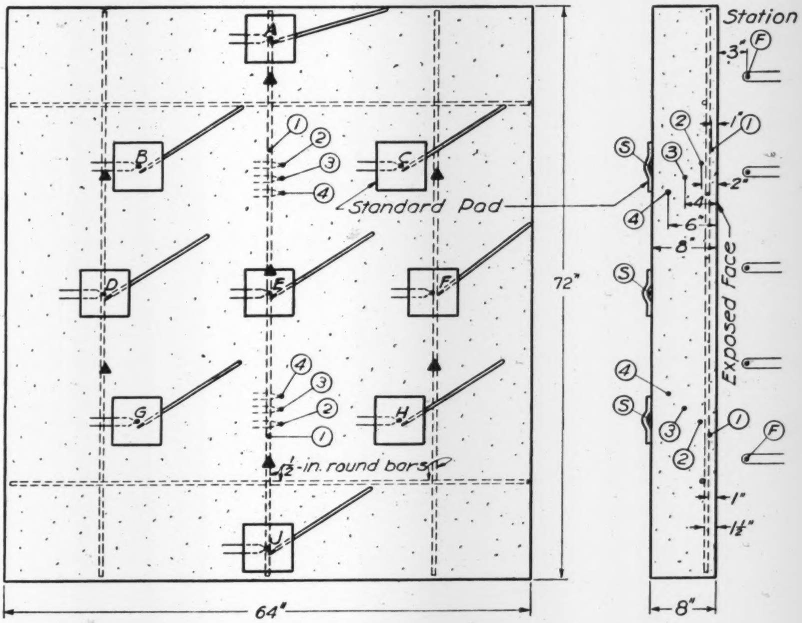

FIG. 1.—Location of Thermocouples and Thermometers for 8-in. Slabs.

The end view of the slab in right diagram indicates the location with respect to the thickness of the slab of thermocouples, *F*, in the furnace combustion chamber, of the thermocouples in the interior of the slab at stations 1, 2, 3, and 4 (1, 2, 4, and 6 in., respectively, from the exposed face), and of thermocouples and thermometers, *S*, under standard pads in contact with the unexposed face of the slab.

The left diagram shows the distribution of thermocouples and thermometers as viewed from the unexposed face of the slab. The locations of the seven furnace thermocouples are indicated by solid triangles. The two groups of thermocouples in the interior of the slab at stations 1, 2, 3, and 4 are indicated by the station numbers. The nine thermocouples and nine thermometers in contact with the unexposed face of the slab and covered by standard pads are lettered *A*, *B*, *C* . . . *J*.

The furnace thermocouples (*F*, right diagram) consisted of No. 8 gage chromel-alumel wire twisted and welded at the sensitive tips. They were all enclosed in sealed "sillimanite" or "mullite" porcelain tubes 1 in. in outside diameter and ⅛-in. in wall thickness. The temperature sensitive point of each couple was placed at a uniform distance of 3 in. from the exposed face of the slab. (This is 3 in. closer than required for walls or partitions by the present A.S.T.M. Standard Methods of Fire Tests of Building Construction and Materials (C 19–41), 1942 Book of A.S.T.M. Standards, Part II, p. 196. The exposed length of the tube and couple varied from 23 to 25 in. depending on the thickness of slab.

The thermocouples in the interior of the slab (stations 1, 2, 3, and 4) consisted of No. 14 B. & S. gage calibrated iron-constantan wire covered with asbestos insulation and twisted and welded at the sensitive tips. They were stripped of insulation for a distance of 3 in. from the welded tip and carefully placed to bring the 3-in. bare portion parallel to the exposed face of the slab at the distance desired. This enhanced the accuracy of the temperature readings at these stations by minimizing the effect of conduction of heat from the sensitive tip to the lead-out wires passing through a mass of comparatively cool concrete.

The thermocouples on the unexposed face of the slab were made of No. 20 B. & S. gage calibrated iron-constantan wires, covered with asbestos insulation, and twisted and welded at the sensitive tips. The wire of this gage was 0.032 in. in diameter which was somewhat finer, and hence more sensitive to temperature changes, than the No. 18 B. & S. gage (0.040 in.) prescribed as the maximum gage by the present Standard C 19–41. The couples were stripped of insulation for a distance of 3½ in. from the welded tips and the bare wires were in all cases held in close contact with the unexposed surface by the standard 6 by 6-in. dry asbestos felt pads 0.4 in. thick.

The thermometers on the unexposed face of the slab were of the standard A.S.T.M. partial-immersion type with stems ⅜ in. in diameter in which the 3-in. long sensitive portions were covered by the same asbestos pads as the thermocouples.

NOTES AND REMARKS FOR TABLE I.

General Notes

The data for the tests are grouped to facilitate study of the effect of different variables. For this reason the data from tests on 3 slabs (116, 118, and 124) appear in two groups.

Materials.—Data on Concrete and Aggregates used in making the slab specimens are given in the notes of Table II.

Curing and Age at Test.—All slabs were cured moist for 5 days (except slabs for tests Nos. 126 and 127 which were cured moist for 10 days), and in the air of the laboratory for the periods given in column 7. These air-drying periods also represent the approximate age of each slab at test except in the case of the slabs for tests Nos. 126 and 127 which were actually 37 and 42 days old, respectively, after the nominal air-drying period of 1 month (actually 27 and 32 days, respectively).

Weight and Moisture Loss Data.—The values given in columns 3 to 6 were determined from small slab specimens 12 in. square as described in the notes accompanying Fig. 3.

Fire-Endurance Period.—Since none of the slabs failed under the applied load during the fire-endurance test and none by the passage of flame and gases hot enough to ignite cotton waste, their fire-endurance periods were determined by an average temperature rise on the unexposed face of 250 F. above initial temperatures. This average rise was attained in all tests before the maximum permissible rise of 325 F. was attained at any of the nine stations where measurements were made with thermocouples and thermometers as described in the notes of Fig. 1.

Measurements of Temperatures of Unexposed Face.—The values in columns 8 and 9 summarize the data on the development of critical end-point temperatures on the unexposed face of the slab. In both columns the values in parentheses are based on thermometer indications whereas those without parentheses are based on corresponding thermocouple indications. The fire endurance periods used in the diagrams of Fig. 2, 4, 11, and 13 are based on the thermocouple indications. The time at which the average rise of 250 F. and the maximum rise of 325 F. was attained was accurately determined to the nearest minute by means of special time-temperature graphs plotted to an enlarged scale. The temperature rise at the end of fire exposure (columns 29 to 31) and after fire exposure (columns 32 to 34) were determined from the time-temperature curves in Figs. 17 to 26. All values of temperature rise and time are reckoned from start of test.

Duration of Exposure.—The exposure to fire was usually continued beyond the time the permissible temperature rise (avg. 250 F., max. 325 F.) had been attained on the unexposed face of the slab being terminated, in general, at the next half-hour period. The outstanding exception to this general procedure was test No. 123. (See note (*b*) under "Remarks.")

Furnace Temperature Control.—The degree of compliance in each test with the standard time-temperature curve is indicated by the curves for furnace temperatures in the diagrams of Figs. 17 to 26. In these diagrams the maximum and minimum temperatures indicated by seven furnace thermocouples (Fig. 1) are represented by the dotted line curves.

Load Carried by Slab.—The values in column 11 are the working loads imposed on the slabs during fire exposure. The values in column 12 refer to the ultimate strength and load-carrying ability of the slabs after the two fire-exposure periods indicated in parentheses. The total load which could be imposed on a slab by the combined thrust of the 4 hydraulic jacks of the loading apparatus was 436,000 lb. For 14 of the 16 slabs tested this load was insufficient to cause failure of the slab. In the two slabs which failed during load application, the unit compressive stress at failure is accompanied by the word "ultimate" (ult.). For the 14 slabs which did not fail under load application the unit load at capacity of the loading apparatus is preceded by the word "over."

Deflections and Expansions of Slab.—Values in columns 14 to 19 are based on initial readings at beginning of each exposure to fire.

Measurement of Temperatures Within Slab.—The average time at which 1000 F. was attained at stations in the interior of the slab (columns 20, 21, and 23) was determined from the time-temperature plots used in preparing the time-temperature curves in Figs. 16 to 26. The values in column 22 for a distance of 3 in. from exposed face were interpolated from the curves for 2 and 4 in. Similar remarks apply to the data for 1100 F. in columns 25 to 28. See notes of Fig. 1 for location and description of thermocouples used for these measurements.

Gas Burned.—Chicago City gas, with a heat value controlled at 800 Btu. per cu. ft., was burned in the test furnace. The rate at which the gas was burned to follow the standard time-temperature control curve was practically constant for any given test regardless of its duration. While the rate at which the gas was burned was practically constant for each test it varied as indicated in column 37. In general somewhat more gas was burned in tests of the more massive gravel slabs than in the case of the lighter Haydite slabs.

Remarks

(Letters in parentheses refer to corresponding letters in columns 8, 9, 12, 20, 21, 22, 25, 26, and 27)

(*a*) All values in columns 8 and 9 and in columns 20, 21, and 22 for test No. 124-R include effects of fusion which began at about 6½ hr. (2190 F.) during first exposure (test No. 124).

(*b*) In test No. 123 the fire exposure was terminated at 9 hr. (540 min.). This was well before the time at which the average rise of 250 F. or the maximum rise of 325 F. could be expected to be attained on the unexposed surface of the slab from the trend of the time-temperature curves. Because of this indication the slab was not withdrawn from the front of the incandescent furnace for cooling in air as were the other slabs. Fusion of the exposed face began at about 6½ hr. The gradual fluxing away with exposure for 9 hr. reduced the thickness of the 8-in. slab about 1¼ to 1½ in. The values at which critical temperatures were attained under these conditions are given in columns 8 and 9 for test 123. However, since these values are higher than would be expected with continued fire exposure, a time of 660 min. instead of 696 min. was used in all comparisons with other slabs. This is considered to be the most likely time at which an average temperature rise of 250 F. would have been indicated by the 9 thermocouples under pads under comparable conditions, and accounts for the use of the value 660 min., or its 11-hr. equivalent, in the diagrams of Figs. 2, 4, 12, and 14.

(*c*) All values in columns 8 and 9 for test No. 123-R include the effects of fusion during first exposure in test No. 123.

(*d*) Load of 850 psi. was accompanied by marked bowing of the slab toward the unexposed face caused by yielding of the weakened concrete on the exposed face.

(*e*) All values in columns 8 and 9 for test 126-R include effects of fusion beginning at about 6½ hr. during first exposure in test No. 126. The extent of fusion was more pronounced than in test No. 124 exposed for 7 hr. instead of 8 hr.

(*f*) Values omitted because of marked fluxing away of concrete on exposed face during first exposure in test No. 123 as described in note (*b*).

(*g*) No values because thermocouples for measurement of the interior temperatures of this slab were cut off prematurely.

TABLE I.—GENERAL DATA OF TESTS FOR FIRE RESISTANCE, THERMAL PROPERTIES, AND STRENGTH OF SOLID CONCRETE SLABS.

Test**	Slab						Time for Unexposed Face of Slab to Attain Temperature Rise Indicated, min.		Duration of Fire Exposure, hr.	Load Carried by Slab, psi.		Compressive Strength of Moist-Cured 6 by 12-in. Concrete Cylinders at 60 Days, psi.
	Thickness, in.	Weight of Concrete, lb. per sq. ft.				Period of Air-Drying, months	Average Rise, 250 F.	Maximum Rise, 325 F.		During Both Fire Exposure Periods	After the Two Fire Exposure Periods Indicated	
		As Cast	Air-Dry at Test	Moisture Loss								
				lb.	Per Cent							
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
EFFECT OF SLAB THICKNESS FOR HIGHLY SILICEOUS SAND AND GRAVEL, GRADED 0 TO ¾-IN.												
No. 121	4	48.7	47.1	1.6	3.4	14	66 (69)	75 (76)	1.5	500	Over 1700	4450
No. 121-R							59 (62)	69 (73)	1.0		(1.5 + 1.0 hr.)	
No. 118	6	71.6	69.6	2.0	2.9	12	150 (157)	164 (171)	2.5	400	Over 1130	4450
No. 118-R							123 (127)	139 (147)	2.0		(2.5 + 2.0 hr.)	
No. 117	8	94.8	91.4	3.4	3.7	13	273 (286)	286 (296)	5.0	400	Over 850	4450
No. 117-R							213 (222)	258 (273)	4.0		(5.0 + 4.0 hr.)	
EFFECT OF SLAB THICKNESS FOR CALCAREOUS SAND AND GRAVEL, GRADED 0 TO ¾-IN.												
No. 119	4	50.2	48.9	1.3	2.7	14	82 (85)	92 (93)	2.0	500	Over 1700	5950
No. 119-R							76 (81)	90 (94)	1.5		(2.0 + 1.5 hr.)	
No. 116	6	75.7	74.1	1.6	2.2	12	196 (205)	224 (220)	3.5	400	Over 1130	5950
No. 116-R							159 (166)	189 (195)	2.5		(3.5 + 2.5 hr.)	
No. 115	8	100.7	98.9	1.8	1.8	13	331 (344)	360 (360)	6.0	400	Over 850	5950
No. 115-R							260 (267)	317 (321)	4.5		(6.0 + 4.5 hr.)	
EFFECT OF SLAB THICKNESS FOR FINE AND COARSE HAYDITE, GRADED 0 TO ¾-IN.												
No. 125	4	33.6	28.9	4.7	16.2	13	140 (147)	151 (155)	2.5	500	750 (Ult.)	3250
No. 125-R							95 (101)	105 (110)	2.0		(2.5 + 2.0 hr.)	
No. 124	6	48.9	43.1	5.8	13.5	13	383 (393)	403 (409)	7.0	400	Over 1130	3250
No. 124-R							201^a (211)	225^a (236)	3.5		(7.0 + 3.5 hr.)	
No. 123	8	67.6	60.1	7.5	12.4	14	*696*^{b} *(732)*	*758*^{b} ...	9.0	400	Over 850^d	3250
No. 123-R							239^c (259)	268^c (300)	4.5		(9.0 + 4.5 hr.)	
EFFECT OF DURATION OF DRYING FOR CALCAREOUS SAND AND GRAVEL, GRADED 0 TO ¾-IN.												
No. 127	6	77.3	76.6	0.7	0.9	1	181 (186)	199 (206)	3.5	400	Over 1130	...
No. 127-R						(32 da.)	151 (156)	172 (182)	2.5		(3.5 + 2.5 hr.)	
No. 116	6	75.7	74.1	1.6	2.2	12	196 (205)	224 (220)	3.5	400	Over 1130	5950
No. 116-R							159 (166)	189 (195)	2.5		(3.5 + 2.5 hr.)	
EFFECT OF DURATION OF DRYING FOR FINE AND COARSE HAYDITE GRADED 0 TO ¾-IN.												
No. 126	6	47.8	46.1	1.7	3.7	1	487 (491)	495 (496)	8.0	400	820 (Ult.)	...
No. 126-R						(27 da.)	197^e (204)	194^e (211)	3.5		(8.0 + 3.5 hr.)	
No 124	6	48.9	43.1	5.8	13.5	13	383 (393)	403 (409)	7.0	400	Over 1130	3250
No. 124-R							201^a (211)	225^a (236)	3.5		(7.0 + 3.5 hr.)	
EFFECT OF SIZE OF HIGHLY SILICEOUS SAND AND GRAVEL*												
No. 120	6	71.9	70.2	1.7	2.4	12	158 (163)	178 (182)	3.0	400	Over 1130	4300
No. 120-R	(0-⅜")						127 (132)	146 (150)	2.5		(3.0 + 2.5 hr.)	
No. 118	6	71.6	69.6	2.0	2.9	12	150 (157)	164 (171)	2.5	400	Over 1130	4500
No. 118-R	(0-¾")						123 (127)	139 (147)	2.0		(2.5 + 2.0 hr.)	
No. 122	6	72.8	70.5	2.3	3.2	12	143 (147)	159 (162)	2.5	400	Over 1130	4800
No. 122-R	(0-1½")						119 (123)	139 (142)	2.0		(2.5 + 2.0 hr.)	
EFFECT OF SIZE OF SILICEOUS GRAVEL* IN MIXTURE WITH EQUAL VOLUME OF 0 TO ¾-IN. HAYDITE												
No. 157	6	59.4	56.0	3.4	6.1	12	239 (252)	256 (264)	4.5	400	Over 1130	4500
No. 157-R	(0-⅜")						164 (172)	188 (195)	3.0		(4.5 + 3.0 hr.)	
No. 158	6	61.3	58.5	2.8	4.8	12	252 (262)	271 (282)	4.5	400	Over 1130	4450
No. 158-R	(0-¾")						177 (185)	204 (213)	3.0		(4.5 + 3.0 hr.)	
No. 159	6	60.4	57.2	3.2	5.6	12	254 (263)	272 (282)	4.5	400	Over 1130	4200
No. 159-R	(0-1½")						172 (180)	199 (207)	3.0		(4.5 + 3.0 hr.)	

* Size range indicated under thickness in column 2.

** Test numbers with letter "R" denote second fire-endurance test.

(Continued on pp. 1106 and 1107)

TABLE I.—*Continued.*

Test**	Thickness, in.	Maximum Lateral Deflection at Center of Slab, in.					Maximum Vertical Expansion, in. per foot of slab	Time to Attain 1000 F. at Indicated Distances from Exposed Face of Slab, min.				Average Initial Slab Temperature at Start of Test, deg. Fahr.
		During Fire Exposure at Time Indicated,		At End of Fire Exposure Period,		At End of Cooling Period,						
		inward	hours	inward	hours	outward		1 in.	2 in.	3 in.	4 in.	
(1)	(2)	(14)	(15)	(16)	(17)	(18)	(19)	(20)	(21)	(22)	(23)	(24)
EFFECT OF SLAB THICKNESS FOR HIGHLY SILICEOUS SAND AND GRAVEL, GRADED 0 TO ¾-IN.												
No. 121	4	1.28	1.1	1.25	1.5	0.09	0.061	78	115	150	...	71
No. 121-R		0.81	1.0	0.81	1.0	0.06	0.040	76	107	...	...	74
No. 118	6	0.63	0.9	0.54	2.5	0.31	0.066	83	184	247	...	71
No. 118-R		0.59	1.9	0.58	2.0	0.03	0.052	92	173	...	...	71
No. 117	8	0.56	1.0	0.29	5.0	0.50	0.064	102	157	260	390	68
No. 117-R		0.50	3.6	0.52	4.0	0.00	0.055	123	193	300	...	73
EFFECT OF SLAB THICKNESS FOR CALCAREOUS SAND AND GRAVEL, GRADED 0 TO ¾-IN.												
No. 119	4	0.53	0.6	0.38	2.0	0.25	0.050	104	157	212	...	71
No. 119-R		0.53	1.3	0.49	1.5	0.03	0.039	100	138	...	...	79
No. 116	6	0.50	0.8	0.44	3.5	0.13	0.051	99	257	360	...	73
No. 116-R		0.47	2.3	0.45	2.5	0.06	0.040	115	227	...	...	71
No. 115	8	0.44	0.8	0.29	6.0	0.31	0.049	101	212	375	545	67
No. 115-R		0.41	3.2	0.41	4.5	0.09	0.038	111	220	360	...	71
EFFECT OF SLAB THICKNESS FOR FINE AND COARSE HAYDITE, GRADED 0 TO ¾-IN.												
No. 125	4	0.19	0.3	0.03 Out	2.5	0.94	0.011	100	180	265	...	73
No. 125-R		0.69	0.8	0.64	2.0	0.37	0.031	81	142	...	...	74
No. 124	6	0.19	0.3	0.23 Out	7.0	0.88	0.014	105	172	325	525	71
No. 124-R		0.50	1.0	0.41	3.5	0.41	0.022	68[a]	103[a]	205[a]	...	74
No. 123	8	0.19	0.4	0.25 Out	9.0	0.88	0.013	127[f]	272[f]	430	570	70
No. 123-R		0.31	1.7	0.14	4.5	0.47	0.022	...	...	...	...	73
EFFECT OF DURATION OF DRYING FOR CALCAREOUS SAND AND GRAVEL, GRADED 0 TO ¾-IN.												
No. 127	6	0.53	0.6	0.41	3.5	0.19	0.050	111	225	...	...	70
No. 127-R		0.47	1.8	0.47	2.5	0.03	0.044	113	195	...	...	86
No. 116	6	0.50	0.8	0.44	3.5	0.13	0.051	99	257	...	...	73
No. 116-R		0.47	2.3	0.45	2.5	0.06	0.040	115	227	...	...	71
EFFECT OF DURATION OF DRYING FOR FINE AND COARSE HAYDITE, GRADED 0 TO ¾-IN.												
No. 126	6	0.16	0.3	0.28 Out	8.0	0.84	0.011	123	275	440	600	66
No. 126-R		0.37	0.8	0.34	3.5	0.22	0.026	...[g]	...[g]	...	...	75
No. 124	6	0.19	0.3	0.23 Out	7.0	0.88	0.014	105	172	325	...	71
No. 124-R		0.50	1.0	0.41	3.5	0.41	0.022	68[a]	103[a]	205[a]	...	74
EFFECT OF SIZE OF HIGHLY SILICEOUS SAND AND GRAVEL*												
No. 120	6	0.56	1.1	0.42	3.0	0.44	0.062	93	165	...	...	70
No. 120-R	(0–⅜")	0.53	2.2	0.53	2.5	0.13	0.058	101	165	...	...	64
No. 118	6	0.63	0.9	0.54	2.5	0.31	0.066	83	184	...	...	71
No. 118-R	(0–¾")	0.59	1.9	0.58	2.0	0.03	0.052	92	173	...	...	71
No. 122	6	0.69	1.2	0.54	2.5	0.28	0.064	77	197	...	...	65
No. 122-R	(0–1½")	0.59	1.9	0.59	2.0	0.00	0.051	90	196	...	...	75
EFFECT OF SIZE OF SILICEOUS GRAVEL* IN MIXTURE WITH EQUAL VOLUME OF 0 TO ¾-IN. HAYDITE												
No. 157	6	0.41	0.5	0.14	4.5	0.47	0.040	98	220	...	...	78
No. 157-R	(0–⅜")	0.50	1.7	0.44	3.0	0.09	0.040	80	180	...	...	86
No. 158	6	0.44	0.6	0.17	4.5	0.50	0.045	83	168	295	...	76
No. 158-R	(0–¾")	0.41	1.4	0.41	3.0	0.03	0.043	76	145	...	...	86
No. 159	6	0.37	0.7	0.08	4.5	0.56	0.038	104	202	315	...	79
No. 159-R	(0–1½")	0.44	1.6	0.43	3.0	0.00	0.039	89	172	...	...	90

* Size range indicated under thickness in column 2.

** Test numbers with letter "R" denote second fire-endurance test.

TABLE 1.—*Continued.*

Test**	Thickness, in.	Time to Attain 1100 F. at Indicated Distances from Exposed Face of Slab, min.				Temperature Rise at End of Fire Exposure, deg. Fahr.			Temperature Rise After Fire Exposure, deg. Fahr.			Date Slab Made	Date Slab Tested	Gas Burned During Test, average, cu. ft. per hr.
						Time, hr.	Couples Under Pads		Time, hr.	Couples Under Pads				
		1 in.	2 in.	3 in.	4 in.		Avg.	Max.		Avg.	Max.			
(1)	(2)	(25)	(26)	(27)	(28)	(29)	(30)	(31)	(32)	(33)	(34)	(35)	(36)	(37)
EFFECT OF SLAB THICKNESS FOR HIGHLY SILICEOUS SAND AND GRAVEL, GRADED 0 TO ¾-IN.														
No. 121	4	97	135	...	...	1.5	379	410	2.3	519	548	Nov. 29, 1930	Jan. 21, 1932	2310
No. 121-R		90	...	...	...	1.0	255	274	1.7	410	432		“ 22, “	2190
No. 118	6	102	217	...	...	2.5	256	288	3.8	407	441	Nov. 24, 1930	Nov. 19, 1931	2060
No. 118-R		113	195	...	...	2.0	244	275	3.3	389	419		“ 21, “	1905
No. 117	8	125	192	310	442	5.0	302	347	7.0	432	455	Nov. 20, 1930	Dec. 16, 1931	2040
No. 117-R		150	238	...	...	4.0	288	301	6.3	399	417		“ 18, “	1900
EFFECT OF SLAB THICKNESS FOR CALCAREOUS SAND AND GRAVEL, GRADED 0 TO ¾-IN.														
No. 119	4	135	187	...	...	2.0	424	441	2.8	531	559	Nov. 25, 1930	Jan. 28, 1932	2255
No. 119-R		117	...	...	...	1.5	311	321	2.3	453	470		“ 29, “	2100
No. 116	6	135	310	...	...	3.5	281	307	5.1	417	440	Nov. 19, 1930	Nov. 13, 1931	2000
No. 116-R		145	227	...	...	2.5	229	239	4.2	367	382		“ 16, “	1890
No. 115	8	137	282	460	622	6.0	293	325	8.3	410	430	Nov. 14, 1930	Dec. 9, 1931	2030
No. 115-R		145	280	...	...	4.5	261	270	6.8	368	379		“ 11, “	1910
EFFECT OF SLAB THICKNESS FOR FINE AND COARSE HAYDITE, GRADED 0 TO ¾-IN.														
No. 125	4	124	217	...	...	2.5	287	324	3.5	432	477	Dec. 11, 1930	Jan. 13, 1932	2020
No. 125-R		100	...	...	...	2.0	348	385	2.9	478	517		“ 15, “	1925
No. 124	6	127	205	380	570	7.0	320	359	8.7	449	488	Dec. 9, 1930	Jan. 6, 1932	1855
No. 124-R		85[a]	126[a]	240[a]	...	3.5	266	296	5.2	398	428		“ 8, “	1860
No. 123	8	155	317	490	600	9.0	159	185	13.3	290	327	Dec. 5, 1930	Feb. 3, 1932	1920
No. 123-R		...[f]	...[f]	...	...	4.5	288	327	6.3	377	416		“ 8, “	1840
EFFECT OF DURATION OF DRYING FOR CALCAREOUS SAND AND GRAVEL, GRADED 0 TO ¾-IN.														
No. 127	6	150	287	...	...	3.5	318	352	4.8	428	469	Dec. 15, 1930	Jan. 26, 1931	2000
No. 127-R		145	...	...	...	2.5	249	272	3.9	373	406		“ 27, “	1775
No. 116	6	135	310	...	...	3.5	281	307	5.1	417	440	Nov. 19, 1930	Nov. 13, 1931	2000
No. 116-R		145	227	...	...	2.5	229	239	4.2	367	382		“ 16, “	1890
EFFECT OF DURATION OF DRYING FOR FINE AND COARSE HAYDITE, GRADED 0 TO ¾-IN.														
No. 126	6	150	320	492	...	8.0	234	294	9.8	396	453	Dec. 13, 1930	Jan. 19, 1931	1780
No. 126-R		...[g]	...[g]	...	...	3.5	275	360	5.0	385	465		“ 21, “	1640
No. 124	6	127	205	380	570	7.0	320	359	8.7	449	488	Dec. 9, 1930	Jan. 6, 1932	1855
No. 124-R		85[a]	126[a]	240[a]	...	3.5	266	296	5.2	398	428		“ 8, “	1860
EFFECT OF SIZE OF HIGHLY SILICEOUS SAND AND GRAVEL*														
No. 120	6	112	200	...	...	3.0	309	330	4.3	440	462	Nov. 28, 1930	Nov. 25, 1931	2130
No. 120-R	(0–⅜″)	125	190	...	...	2.5	316	336	3.8	436	462		“ 27, “	2000
No. 118	6	102	217	...	...	2.5	256	288	3.8	407	441	Nov. 24, 1930	Nov. 19, 1931	2060
No. 118-R	(0–¾″)	113	195	...	...	2.0	244	275	3.3	389	419		“ 21, “	1905
No. 122	6	95	230	...	...	2.5	270	297	3.8	430	455	Dec. 4, 1930	Dec. 2, 1931	2150
No. 122-R	(0–1½″)	112	...	...	...	2.0	255	270	3.4	395	425		“ 3, “	2060
EFFECT OF SIZE OF SILICEOUS GRAVEL* IN MIXTURE WITH EQUAL VOLUME OF 0 TO ¾-IN. HAYDITE														
No. 157	6	120	265	...	...	4.5	314	362	6.1	434	470	Aug. 5, 1931	Aug. 15, 1932	1970
No. 157-R	(0–⅜″)	100	210	...	...	3.0	284	312	4.5	398	434		“ 17, “	1910
No. 158	6	102	205	355	...	4.5	285	324	6.1	424	452	Aug. 7, 1931	Aug. 22, 1932	1895
No. 158-R	(0–¾″)	93	180	...	...	3.0	254	274	4.5	384	404		“ 24, “	1780
No. 159	6	127	243	362	...	4.5	285	321	6.2	431	455	Aug. 10, 1931	Aug. 29, 1932	1850
No. 159-R	(0–1½″)	111	210	...	...	3.0	265	280	4.5	390	415		“ 31, “	1805

* Size range indicated under thickness in column 2.

** Test numbers with letter “R” denote second fire-endurance test.

Two thermocouples were used to obtain temperatures at each distance from the exposed face. These couples were placed across the vertical center line of the slab as shown in Fig. 1 so that they indicated temperatures in the concrete near the center of the upper and lower halves of the slab. For further details see notes for Fig. 1.

TABLE II.—DATA ON CONCRETE AND AGGREGATES USED IN SOLID SLABS.

Concrete: Made from a constant mix (1:5 by dry rodded volume, net water-cement ratio 6.5 to 7.0 gal. per sack cement) and the three widely different aggregates described below. Cement content 5.2 sacks (about 489 lb.) per cu. yd.; plastic consistency (5 to 6-in. slump) placed by hand rodding. Cement was a laboratory mixture of equal parts by weight of four commercial brands of normal strength portland cement (present A.S.T.M. Type 1) purchased in Chicago.

Description of Aggregates:

(1) *Siliceous Sand and Gravel.*—A highly siliceous aggregate (Meramec River, Mo.) with 95 per cent silica in form of flint and chert. Particles very irregular with both sharp and slightly rounded edges and both polished and pitted surface.

(2) *Calcareous Sand and Gravel.*—A calcareous aggregate (Elgin, Ill.) containing about 40 per cent calcium carbonate, 30 per cent magnesium carbonate, and less than 15 per cent quartz. Particles range from regular to irregular in shape but generally with well-rounded edges. Surfaces varied from smooth, slippery texture to a rough, porous, pitted type with neither type predominating.

(3) *Fine and Coarse Haydite.*—A light, porous, aggregate material (Western Brick Co., Danville, Ill.) prepared by burning shale to incipient fusion and crushing and grading the resulting clinker. Particles irregular in shape with slightly rounded, sharp edges and slightly rough, porous and pitted surfaces.

(4) Mixture of equal volumes of siliceous aggregate (1) and Haydite aggregate (3) as indicated.

Test	AGGREGATE								Compressive Strength of Moist-Cured 6 by 12-in. Concrete Cylinders at 60 days, psi.
	Type	Size Range	Fineness Modulus (F.M.)	Dry Rodded Weight, lb. per cu. ft.	Per Cent by Weight of Each Sieve Size in Mixture (Tyler Standard Sieves)[a]				
					0 to ¼ in.	¼ to ⅜ in.	⅜ to ¾ in.	¾ to 1½ in.	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
TESTS WITH INDIVIDUAL AGGREGATES OF SIZE AND GRADING INDICATED									
Nos. 121, 121-R Nos. 118, 118-R Nos. 117, 117-R	Siliceous gravel	0 to ¾ in.	4.6	121	47 (F.M. 2.5; Wt. 107)	20 (F.M. 5.9; Wt. 93)	33 (F.M. 7.0; Wt. 93)	...	4450
Nos. 119, 119-R Nos. 116, 116-R Nos. 115, 115-R Nos. 127, 127-R	Calcareous gravel	0 to ¾ in.	4.9	126	47 (F.M. 3.0; Wt. 113)	18 (F.M. 6.1; Wt. 105)	35 (F.M. 7.0; Wt. 105)	...	5950
Nos. 125, 125-R Nos. 124, 124-R Nos. 123, 123-R Nos. 126, 126-R	Haydite	0 to ¾ in.	3.8	63	70 (F.M. 2.7; Wt. 59)	18 (F.M. 6.0; Wt. 46)	12 (F.M. 7.0; Wt. 43)	...	3250
Nos. 120, 120-R	Siliceous gravel	0 to ⅜ in.	4.3	117	47	53	...	...	4300
Nos. 118, 118-R		0 to ¾ in.	4.6	121	47	20	22	...	4500
Nos. 122, 122-R		0 to 1½ in.	4.9	123	47	13	27	13[b]	4800
MIXTURES OF EQUAL VOLUMES OF SILICEOUS AGGREGATE OF SIZE RANGE INDICATED WITH 0 TO ¾-IN. HAYDITE									
Nos. 157, 157-R	Sil. gravel	0 to ⅜ in.	4.3	117	47	53	...	...	4500
	Haydite	0 to ¾ in.	3.8	63	50	70	18	12	
Nos. 158, 158-R	Sil. gravel	0 to ¾ in.	4.6	121	47	20	33	...	4450
	Haydite	0 to ¾ in.	3.8	63	50	70	18	12	
Nos. 159, 159-R	Sil. gravel	0 to 1½ in.	4.9	123	47	13	27	13	4200
	Haydite	0 to ¾ in.	3.8	63	50	70	18	12	

[a] Values in parentheses in columns 6, 7, and 8 represent the fineness modulus (F.M.) and dry rodded weight (lb. per cu. ft.) of each sieve size. These values are included only once for any given size and type of aggregate.

[b] Fineness modulus, 8.0; dry rodded weight, 88 lb. per cu. ft.

(*d*) *Measurement of Temperature of Unexposed Face.*—Temperatures on the unexposed face of the slab were obtained at nine locations by both thermocouples and thermometers under standard asbestos pads as shown in Fig. 1. From these temperatures the times to attain the average rise of 250 F. and the maximum permissible rise of 325 F. above initial slab temperature, as defined by the standard fire test specifications, were determined. For further details see notes for Fig. 1.

(*e*) *Intensity and Period of Fire Exposure.*—The slab while under load was exposed on one side to gas flames of controlled intensity in accordance with a predetermined time-temperature curve designated in Figs. 17 to 26 as the "standard furnace control curve." It is identical with the time-temperature curve required by the standard fire test specifications. The fire exposure period was usually continued to the nearest half hour after the attainment of the critical end-point temperatures on the unexposed face of the slab described in (*d*) above.

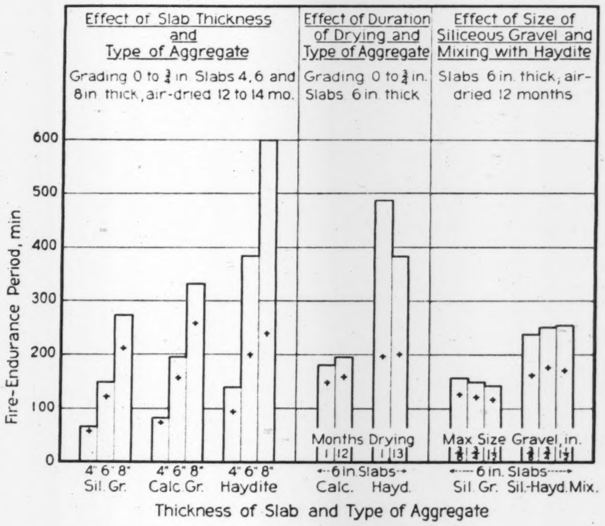

FIG. 2.—Fire-Endurance Period for First and Second Exposure to Fire of Solid Concrete Slabs

Heights of columns represent time (fire-endurance period) required to attain average temperature rise of 250 F. on unexposed face during first exposure to fire. Plotted crosses indicate time required to attain this same temperature rise during second exposure to fire.

Data from Table I (column 8).

Furnace temperatures were indicated by seven calibrated thermocouples symmetrically placed at various stations on the exposed slab area as shown in Fig. 1. For further details see notes for Fig. 1.

(*f*) *Loading of Slabs.*—Just before the first exposure to fire a load of 500 psi. was applied (as a load-bearing wall) to the 4-in. slabs, and a load of 400 psi. to the 6 and 8-in. slabs, by means of hydraulic jacks. These loads were maintained during the entire period of fire exposure and in the subsequent period of cooling to room temperature. The same loads were also maintained during the second fire exposure and cooling period. All slabs were then loaded to their ultimate strength or to the maximum capacity of the hydraulic jacks which totaled about 436,000 lb. This capacity of equipment was equivalent to about 1700, 1130, and 850 psi., respectively, for slabs 4, 6 and 8 in. thick.

(*g*) *Observations During Test and Cooling Period.* During exposure to fire and during the subsequent cooling period both the vertical expansion and the lateral deflection or bowing of the wall were measured. A record was made of the appearance of moisture, development of cracks, fusion or other effects of fire exposure or of cooling after fire exposure. A photographic record was made of the appearance of each slab at various stages of the test.

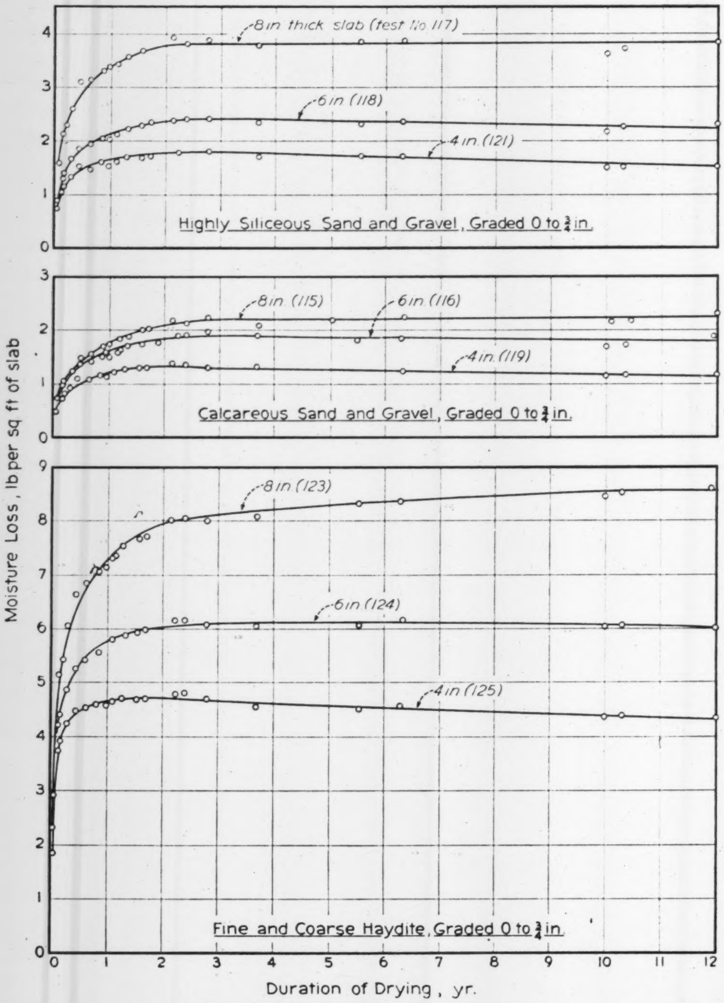

FIG. 3.—Moisture Loss of Small Slab Specimens During Storage in Air of Laboratory.

Each specimen was cast in a steel mold 12 in. square to the same thickness and from the same concrete as the 5⅓ by 6-ft. slab exposed to fire. After curing in the mold for 5 days the four edges of the specimen were carefully sealed with paraffin. Thereafter the specimens were stored on one edge in a rack so that the two opposite vertical faces (each 12 in. square) were exposed to the freely circulating air of the laboratory. During the winter months the temperature was maintained at 70 to 75 F. and the relative humidity corresponded to the low percentages common in a heated building. Changes in weight were determined with a platform scale sensitive to 0.01 lb.

Data of Tests:

The principal data of the tests on the 16 solid slabs are given in Table I. These are arranged in groups to facilitate study of the effect of different variables. Pertinent information is given in the General Notes and Remarks accompanying this table. Information on the concrete and on the aggregates used in the solid slabs is given in the notes and data of Table II. Details and results of the tests of the two rib-type slabs are given in Table IV and the notes accompanying this table.

The significant results of the slab tests are presented graphically in Figs. 2 to 4, 6, 7, 12 to 14, and 16. Data on the properties of carbon steel at elevated temperatures assembled from various sources are presented in Figs. 8 to 11.

The time-temperature curves for the first and second fire-endurance test of each solid slab specimen are given in Figs. 17 to 26 of the Appendix. So far as possible these diagrams are grouped to follow the order of tests given in Table I.

The different phases of the investigation are discussed under the following headings:

I. Performance of Solid Slabs as Bearing Walls,
II. Thermal Properties of the Solid Slabs and Their Practical Significance in the Design of Reinforced Concrete Floors,
III. Results of Tests on Bare and Plastered Ribbed-Slab Specimens and Their Significance,
IV. Conclusions.

I. PERFORMANCE OF SOLID SLABS AS BEARING WALLS

Comparisons of the performance of the various test slabs are based principally on two criteria:

1. Fire-endurance period, and
2. Strength or load-carrying ability (tested as walls) after fire exposure.

Fire-Endurance Period.—This is the period during which the test slabs carried the applied load (500 psi. for 4-in. slabs, 400 psi. for 6 and 8-in. slabs) under standard fire exposure, without transmission of flame, hot gases, and high temperatures to the unexposed side as defined by the standard fire test specifications. Since none of the slabs failed under the applied load during the fire-endurance test and none by the passage of flame and gases hot enough to ignite cotton waste, their fire-endurance periods were determined by the temperature rise on the unexposed face of the slab. The requirements of the standard are that the average temperature rise (above initial specimen temperatures) of the unexposed face under standard asbestos pads shall not exceed 250 F. and that the maximum rise at any point where temperature measurements are taken shall not exceed 325 F. For all tests herein reported the fire-endurance period is based either on the time at which nine No. 20 B. & S. gage calibrated iron-constantan thermocouples symmetrically distributed and in contact with the unexposed face under standard pads indicated an average rise of 250 F. or a rise at any individual point of 325 F. In all of the tests on solid slabs the average rise was attained before the maximum permissible rise at any point.

Although the exacting test technique followed gives a reliable basis for comparison of the *relative* fire-endurance periods of the different wall slabs, it should be pointed out that the fire-endurance periods obtained are at least 8 per cent lower than would be obtained in classification tests of walls 10 by 10 ft. in size but otherwise identical under the requirements of the present specifications. The reasons for this difference are discussed under "Comments on Values of Fire-Endurance Period" in the Appendix to this paper.

Strength After Fire Exposure.—The standard specification does not require that bearing walls subjected to fire-endurance tests carry loads after fire exposure greater than the working loads. However, ability to carry greater loads is a desirable property and provides evidence as to the safety of walls during and after fire exposure and the extent of the fire damage. Therefore, each slab, after cooling to room temperature following the second exposure to fire, was either loaded to failure or was subjected to as much load as could be applied with the test equipment used.

EFFECT OF VARIOUS FACTORS ON THE FIRE ENDURANCE OF SOLID SLABS

Thickness of Slab (left diagram Fig. 2):

The fire-endurance period increased greatly with increase in thickness. In general, for each type of aggregate, the fire-endurance period was doubled when the slab thickness was increased 35 to 40 per cent.

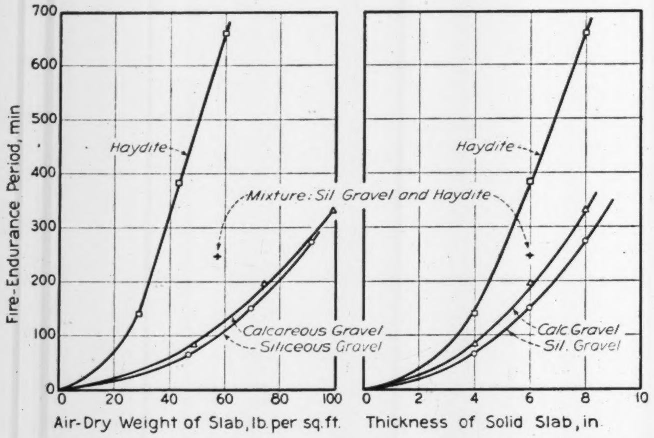

FIG. 4.—Relationships Between Weight per Square Foot of Slab, Thickness of Slab and Fire-Endurance Period for Different Aggregates.

Data from Table I (Columns 4 and 8).

In estimating probable fire-endurance periods of standard 10 by 10 ft. walls under the requirements of present A.S.T.M. standard methods, the fire-endurance periods taken from the diagrams in this figure should be increased by 8 per cent for the reasons discussed under "Comments on Values of Fire-Endurance Period" in the Appendix to this paper.

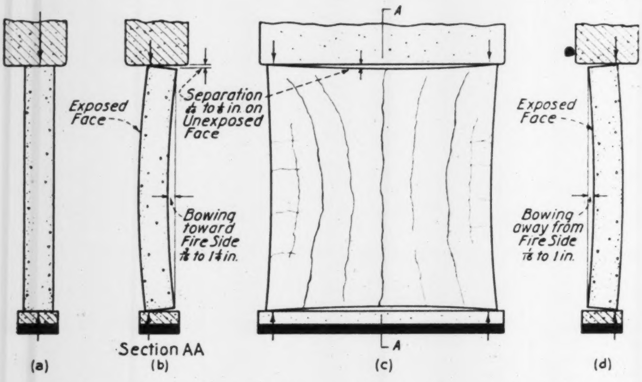
Exposed Face
Separation $\frac{1}{16}$ to $\frac{1}{4}$ in. on Unexposed Face
Bowing toward Fire Side $\frac{3}{16}$ to $1\frac{1}{4}$ in.
A
A
Exposed Face
Bowing away from Fire Side $\frac{1}{16}$ to 1 in.
Section AA
(a)
(b)
(c)
(d)

FIG. 5.—Diagram Illustrating Changes in Loading and in Shape of Wall.

The diagrams show to an exaggerated scale how the uniformly distributed axial loading before fire exposure was changed to a more or less concentrated and eccentric loading with changes in shape and dimensions of wall during and after fire exposure.

Type of Aggregate (left diagram Fig. 2):

For a given slab thickness, the fire-endurance period for the highly siliceous aggregate was about 20 per cent lower and for Haydite about 90 per cent higher than for the calcareous aggregate.

Duration of Drying (center diagram Fig. 2):

The fire-endurance period of a 6-in. Haydite slab after seasoning for 13 months in the air of the laboratory was about 27 per cent less than that of a comparable Haydite slab seasoned for 1 month. This difference is the result of the marked loss of moisture from the highly absorbent porous Haydite concrete during the long seasoning period. With less moisture available for the absorption of heat, the critical end-point temperatures determining the fire-endurance period were attained in less time.

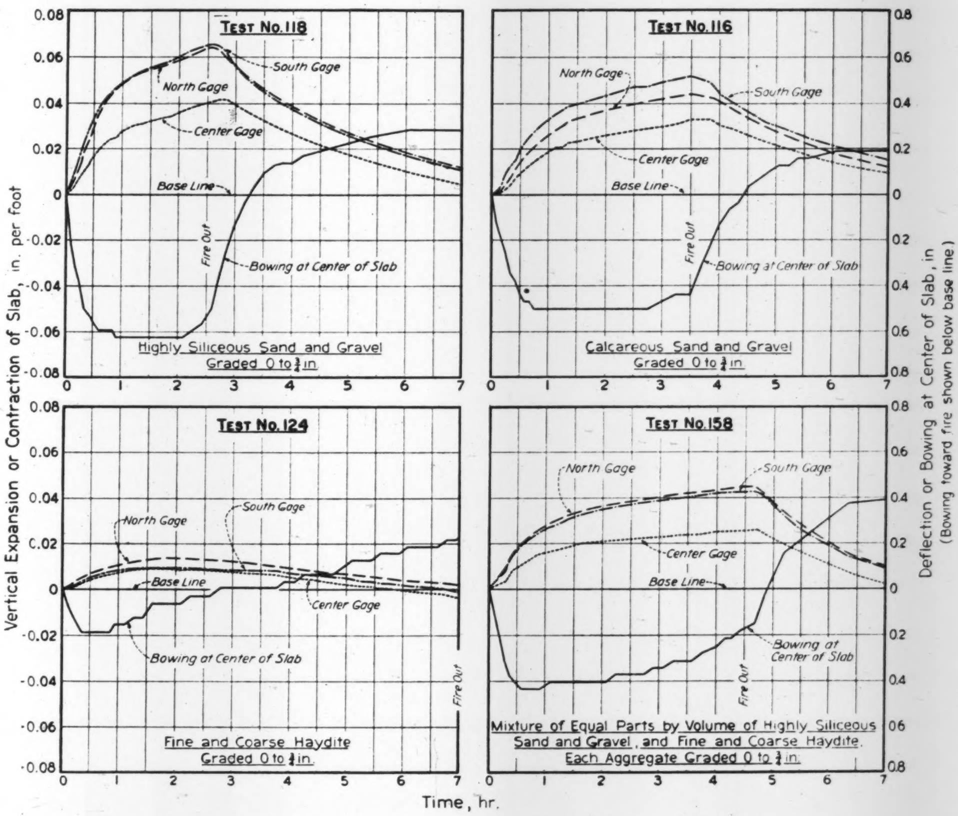

FIG. 6.—Expansion and Deflection Curves of 6-in. Slabs Made with Different Aggregates.

On the other hand, the fire-endurance period of a 6-in. slab of calcareous aggregate seasoned for 1 yr. was about 8 per cent higher than that of a comparable slab seasoned for 1 month. The increase in this case is believed to be due primarily to a more complete hydration of the cement during the longer seasoning period which caused a larger amount of heat to be absorbed during the dehydration that occurred during the fire exposure. In general, the tests indicate that seasoning for 1 yr. is sufficient to provide reliable information on the relative fire endurance and thermal properties of solid concrete slabs.

The curves of Fig. 3 reveal the rate and extent of the loss of moisture from the small auxiliary slabs during storage in the air of the laboratory. Approximately 80 to 90 per cent of the total moisture loss had occurred by the end of the first year. From this it appears that the moisture content of the concrete in the large slab specimens which were seasoned for 12 to 14 months before fire exposure, was not far from the values attained after years of service in a heated building. The 8-in. Haydite slab probably constitutes an exception to this general statement.

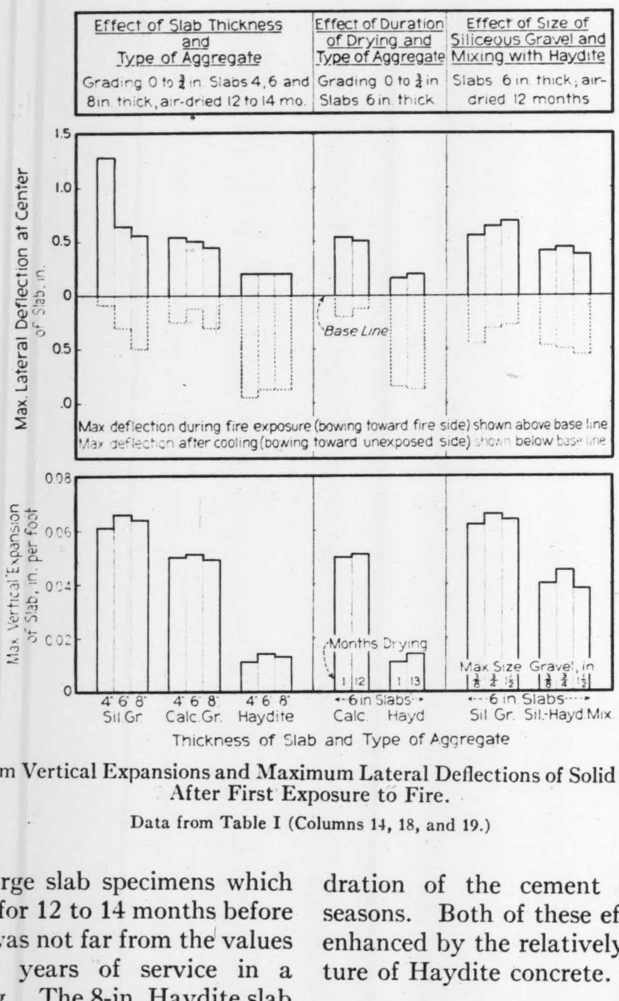

FIG. 7.—Maximum Vertical Expansions and Maximum Lateral Deflections of Solid Slabs During and After First Exposure to Fire.

Data from Table I (Columns 14, 18, and 19.)

Although the curves for the 4-in. and 6-in. slabs for all three aggregates show a fairly rapid and continuous moisture loss until maximum losses were attained, they then showed a slow but gradual increase in weight which was greatest for the 4-in. slabs, particularly with Haydite. This increase in weight may have resulted from carbonation of the lime in the concrete, and from a continued hydration of the cement during humid seasons. Both of these effects would be enhanced by the relatively porous structure of Haydite concrete.

Size of Highly Siliceous Gravel (right diagrams Fig. 2):

For the tests with highly siliceous aggregate of different gradings the fire-endurance period showed a decrease of about 10 per cent when the range of particle size was increased from 0 to $\frac{3}{8}$-in. to 0 to $1\frac{1}{2}$-in. (fineness modulus increase 4.3 to 4.9). This result was similar to that secured in the earlier investigation with tamped concrete masonry units of relatively dry consistency.

Mixing Highly Siliceous Gravel with Equal Volume of Haydite (right diagrams Figs. 2 and 7):

The slabs with siliceous aggregate in combination with an equal volume of Haydite had fire endurance, expansion, deflection and also cracking properties practically intermediate between those of concrete made from each of the aggregates separately. The results compared quite favorably with concrete made from calcareous sand and gravel. Although the weight of the dry concrete from this mixture was about 21 per cent less than that of calcareous sand and gravel alone, the fire endurance period was 28 per cent longer, and the expansion, deflection, and cracking were somewhat less.

Second Exposure to Fire:

The position of the plotted crosses in Fig. 2 relative to the height of the columns indicates that the fire-endurance period of a given slab was always lower in the second exposure to fire than in the first exposure. In the first exposure the temperature transmission was retarded by the heat absorbed by the partial dehydration of the cement and by the vaporization of the moisture in the concrete. Since these two effects were practically absent in the second exposure, the fire-endurance period was determined by the relatively simple thermal properties of a mass of dry, inert material. The over-all effect of the free and combined moisture on the thermal properties of a slab during the first exposure is well illustrated by comparing the curves in Fig. 17 for test No. 118 (first exposure) with the curves for test No. 118-R (second exposure) of a 6-in. slab of siliceous aggregate. For test No. 118 curves 2, 3, and 5 clearly reveal the characteristic lag in temperature rise due to the vaporization of the moisture (principally free moisture) in the slab. The corresponding curves for test No. 118-R, however, show a continuous rise in temperature to the maximum values with none of the intermediate moisture lag effects that are so apparent in the curves for the first exposure. Similar results are shown by comparing the time-temperature curves for the tests of the other slabs represented in the various diagrams of Figs. 17 to 26.

TABLE III.—DIFFERENCE IN FIRE-ENDURANCE PERIOD BETWEEN FIRST AND SECOND EXPOSURE TO FIRE.

Test	Thickness of Slab, in.	Type of Aggregate	Fire-Endurance Period			
			First Exposure, min.	Second Exposure, min.	Reduction (Col. 4 to Col. 5)	
					min.	per cent
(1)	(2)	(3)	(4)	(5)	(6)	(7)
No. 121	4	Siliceous	66	59	7	10.6
No. 118	6		150	123	27	18.0
No. 117	8		273	213	60	22.0
No. 119	4	Calcareous	82	76	6	7.3
No. 116	6		196	159	37	19.0
No. 115	8		331	260	71	21.3
No. 125	4	Haydite	140	95	45	32.1
No. 124	6		383	201	182	48.0
No. 123	8		660	239[a]	421[a]	64.0[a]

[a] This value influenced by the marked fluxing away during the first exposure which reduced the thickness of the 8-in. slab about 1¼ to 1½-in. See note (*b*) under Remarks in the notes for Table I.

The left diagram of Fig. 2 indicates that for a given aggregate the reduction in the fire-endurance period with second exposure increased with the thickness of the slab and that for a given thickness the Haydite slabs show the greatest reduction. This is more clearly shown by the values in column 7 of Table III. These values differ mainly because of inherent differences in the moisture content of slabs of different thickness and type of aggregate after air-seasoning for 12 to 14 months. However, at any period of exposure to drying the thicker slabs will always have a greater percentage of moisture than the thinner slabs. More moisture will be retained at final equilibrium in the thicker slabs because hydration could continue for a longer period. This moisture is held tenaciously in the interior of the slab and can only be removed by unusual methods.

Relationships Between Weight and Thickness of Slab, and Fire-Endurance Period:

The results of the fire-endurance tests are summarized by the diagrams of Fig. 4. The relationships shown are believed to be generally representative of the thermal properties of plastic concrete made with the types of natural and artificial aggregate described in the notes and data of Table II. Although they are based on concrete in which the aggregate was graded from 0 to $\frac{3}{4}$-in. and the cement content was maintained at 5.2 sacks per cu. yd. the fire-endurance period (for a given weight or thickness) would probably be altered less than ±5 per cent by the use of finer or coarser gradings, or by the use of richer or leaner mixes.

The relationship between the air-dry weight of slab and the fire-endurance period in the left diagram of Fig. 4 is similar to the relationships obtained in tests of walls of hollow concrete masonry units [see upper diagrams of Fig. 8 of reference (**17**)]. Comparison of these results indicates that for a given weight the concrete made with the two natural aggregates was from 15 to 30 per cent *less effective* in slabs than in the hollow masonry units whereas the Haydite concrete was from 10 to 40 per cent *more effective* in the slabs than in the units.

LOAD-CARRYING ABILITY OF SOLID SLABS

The repeatedly demonstrated load-carrying ability and safety of the slabs during and after severe fire exposure was one of the important results of these tests. All of the slabs carried the applied load (500 psi. for 4-in. slabs, 400 psi. for 6- and 8-in. slabs) during each of the two fire exposures and subsequent cooling periods with an ample margin of safety (columns 11 and 12, Table I). The ultimate strength of the two slabs which failed during the application of excess load after two fire exposures was 750 and 820 psi. (4-in. Haydite slab, test 125; 6-in. Haydite slab, test 126). The ultimate strength of the other 14 slabs was beyond the capacity of the loading apparatus or over 1130 psi. for 6-in. slabs and over 850 psi. for 8-in. slabs. These loads were carried notwithstanding the changes in shape of slab, the eccentricity and concentration of loading, and other effects of fire exposure and subsequent cooling described later.

The ultimate strength of solid slabs 10 or 11 ft. high but of the same thickness as the 6-ft. high slabs used in these tests could be expected to be somewhat lower (because of a greater slenderness ratio). An evaluation of the strength of large walls after fire exposure was presented in connection with the performance of walls of hollow concrete masonry units [pages 55 to 58 of reference (**17**)]. From that discussion it appears that the strength of large walls or slabs can conservatively be taken as 75 per cent of the strength of the corresponding small slabs used in these tests, after a similar fire exposure. Applying this percentage to the results of tests on the 6-ft. slabs, the ultimate strength of slabs 10 or 11 ft. high after severe fire exposure could be expected to be over 850 psi. for slabs 6 in. thick, and over 650 psi. for slabs 8 in. thick.

EFFECTS OF FIRE EXPOSURE

The exposure of one face of the slabs to fire for periods ranging from 1.5 to 9 hr. and to maximum temperatures of from 1800 to 2400 F. produced changes in the physical properties and composition of the exposed materials and imposed conditions which constituted a severe test of the load-carrying ability of the slabs.

Bond Between Aggregate and Cement Paste:

One effect was to reduce the strength of the concrete and the bond between the cement paste and the aggregate by the gradual dehydration of the cement, and by the expansion and changes in the physical properties of the aggregate. The highly siliceous aggregates showed the greatest thermal expansion and loss of bond.

Spalling and Shattering:

There was no spalling of the concrete either during or after fire exposure in any of the slabs. With slabs of the highly siliceous aggregate it was found that the coarse aggregate particles (from $\frac{1}{4}$-in. up) were shattered in thin planes parallel to the exposed face for a depth of 1 to 1.5 in. This materially weakened the bond between the exposed face and the body of the slab and although the weakened layers remained in position in the vertical slab they might have fallen off in a horizontal slab unless held by the special metal ties sometimes used for highly-siliceous aggregate. The shattering of the aggregate particles probably interrupted the continuity of heat flow from the exposed face to the interior of the slab during the first as well as the second exposure to fire. This effect would be expected to be most pronounced in the 8-in. thick slabs as a result of the longer exposure to fire.

The author has never observed violent disruptive effects in moderately dry concrete during standard fire exposure. However, explosive disruptions have been observed in which sections of the face of *damp* concrete were thrown off within 10 min. from the start of standard fire exposure. This spalling occurred under otherwise favorable conditions as the concrete slabs were made with crushed limestone and no external restraints or stresses due to superimposed loads were involved. Under these conditions the spalling is believed to be the direct result of the building up of steam pressures which could not be released fast enough by diffusion through the dense concrete. With an aggregate having the shattering charcteristics exhibited by the highly siliceous gravel the effect of moisture would be expected to be even more pronounced than with the more stable limestone, particularly if the expansion of the concrete were restrained.

Even if a slab is not restrained externally a substantial restraint may be developed when the fire exposure is localized over small areas instead of being well distributed over the exposed face as prescribed for standard fire tests. This effect would be most severe with the highly expansive aggregates and might cause popping in relatively dry concrete.

It is believed that concretes made with Haydite aggregate, having substantially lower thermal expansion and modulus of elasticity than concretes made with dense natural aggregates, will not develop spalling even when they are appreciably damp and restrained at time of fire exposure. The porous nature of the Haydite concrete precludes the building up of high steam pressures, and its low thermal expansion and modulus of elasticity preclude the development of high stresses during fire exposure.

Calcination:

Although the particles of calcareous aggregate showed no shattering they were definitely softened by calcination with exposure to high temperatures. This calcination retarded the transmission of heat in the high-temperature zone. As pointed out by Ingberg (7) calcination involves a change from the carbonate to the oxide which requires some 430 cal. per g. for calcium carbonate and 340 cal. per g. for magnesium carbonate. The calcined material itself possesses good heat-insulating properties and probably reduced the transmission of heat during both fire exposures and particularly for the 8-in. thick slabs.

Fusion:

The exposed face of the Haydites slabs was but slightly affected by softening during exposure to fire up to 6½ hr. when fusion became evident. Upon exposure to fire for 9 hr. the fluxing which occurred reduced the thickness of the 8-in. slab (test No. 123) about 1¼ or 1½ in. The fluxing away of the exposed face of the relatively dense plastic Haydite concrete appeared to be definitely more pronounced than in corresponding tests of hollow concrete masonry walls in which the units had face shells only 1 in. thick and were made with lean porous Haydite concrete.

Change of Shape of Slab:

Differential expansion between the exposed and unexposed faces caused the slabs to bow toward the fire side as illustrated in Fig. 5. The bowing started during the first 5 min. of fire exposure and usually attained the maximum value during the first hour of exposure. The bowing then either remained constant or decreased gradually as the wall adjusted itself. The maximum deflections (measured at center of unexposed face) varied from about $\frac{3}{16}$ to 1¼ in.

Accompanying the bowing of the slab was a separation between the curved edges at the top of the slab and the straight bearing edge of the top of the test frame (Fig. 5 (*c*)). The maximum separation (measured at the vertical center line on the unexposed face) varied from about $\frac{1}{64}$ to ⅛ in. No corresponding separation developed at the bottom of the slab because the loading beam bent under the thrust of the four hydraulic jacks and conformed to the curved shape of the lower bearing edge.

The diagrams of Fig. 6 illustrate the development of expansion and deflection of the 6-in. slabs during fire exposure. Each diagram represents a slab made with a different aggregate but having the same thickness and the same size range of aggregate. The diagrams also show the contraction and deflection of the slabs during the early part of the cooling period. Curves similar to the ones illustrated were plotted for each test and the essential data are given in columns 14 to 19 of Table I.

From the upper diagram of Fig. 7, which summarizes the data regarding the maximum deflection of each slab during and after the first fire exposure, it is seen that the Haydite slabs deflected much less than slabs of the two natural aggregates during fire exposure, but showed markedly greater bowing toward the unexposed side during the cooling period. This indicates that the Haydite slabs were probably more stable under load during fire exposure than the slabs of natural aggregate but less stable during the cooling period.

The lower diagram of Fig. 7 summarizes the data on the maximum vertical expansion of the slabs during the first exposure to fire. The maximum expansion varied substantially with the type of aggregate but appeared to be practically independent of the range in slab thickness and size of aggregate represented in the tests. The expansion per foot of wall (column 19, Table I) averaged about 0.065 in. for the highly siliceous aggregate, 0.050 in. for the calcareous aggregate, and 0.012 in. for the Haydite.

Cracking:

Of the major cracks which formed on the unexposed face of a slab about half appeared during the first 30 min. of fire exposure but the other half formed more gradually during the next 60 or 90 min. Most of the cracks were vertical, those away from the center curving toward the nearest upper and lower corners of the slab. The few horizontal cracks which formed usually extended for short distance from each vertical edge of the slab to the nearest major vertical crack. The general pattern of cracking is illustrated by Fig. 5 (*c*).

For a given aggregate, the number and width of the cracks increased with the thickness of the slab. The Haydite slabs showed definitely less cracking than corresponding slabs of natural aggregate. The number of cracks was about the same for slabs of calcareous aggregate as for siliceous aggregate but they were generally narrower and did not appear to be as deep.

No cracks were visible on the exposed face during fire exposure, but they became visible after several hours of cooling and, in general, were found to coincide with the major cracks on the unexposed face. Other smaller cracks formed near the corners of the slabs where the greatest load concentration occurred (Fig. 5 (*c*)) when the slab changed shape due to differential expansion.

Change in Character of Loading with Bowing of Slab:

It will be apparent from Fig. 5 that the uniformly distributed axial loading applied before fire exposure (diagram *a*) changed to a more or less concentrated and eccentric loading (diagrams *b* and *c*) with changes in shape and dimensions of the slab during fire exposure. It is obvious, therefore, that for a greater part of the test period the hot material on the exposed face of the slab had to resist compressive stresses substantially greater than would normally result from a load of 400 or 500 psi. uniformly distributed over the potential bearing area.

With the readjustments that took place during cooling, the bowing of the slab toward the exposed face decreased (see curves of Fig. 6) and eventually the slab was bowed toward the unexposed face as shown by Fig. 5 (*d*). The latter bowing was permanent and maximum deflections of from about $\frac{1}{16}$ to 1 in. toward the unexposed face were recorded when the slabs had cooled to room temperature. This produced a shifting of a considerable proportion of the load applied by the hydraulic jacks toward the unexposed portion of the slab. Hence, at the end of the first cooling period and at the beginning of the second exposure to fire, the unexposed portion of the slab carried most of the applied load. The load shifted as the slab bowed toward the fire side during the second exposure and shifted again as the slab bowed away from the fire side during the second cooling period. The fact that all of the slabs carried the applied loads, under the adverse conditions of two exposure and cooling periods, adds to the significance of the data that have been presented regarding their general load-carrying ability after the second exposure to fire.

II. THERMAL PROPERTIES OF SOLID SLABS AND THEIR PRACTICAL SIGNIFICANCE IN THE DESIGN OF REINFORCED CONCRETE FLOORS

Considerations in Extension of Results to Floor Slabs:

Although the slabs were tested in a vertical position to determine their performance as bearing walls, the data on temperature rise on the unexposed face and at various points in the interior of the slab established some general relationships which apply not only to vertical slabs but also to horizontal and inclined slabs (fire exposure under slab). Since these relations are useful in several ways their practical significance with respect to the design of reinforced concrete floor slabs for given fire-endurance periods will be pointed out.

Under the standard fire test specification the fire-endurance period of floor constructions is defined in the same manner as that for load-bearing walls except that the under side of the specimen is exposed to fire. The fire-endurance period is reached when:

(1) An average temperature rise of 250 F. or a maximum rise of 325 F. measured with thermocouples under asbestos pads, is attained on the unexposed side (upper side) of the construction;

(2) Passage of heat, flame, or gases hot enough to ignite cotton waste occurs; or

(3) Structural collapse under the design load occurs.[8]

It will be obvious that structural collapse of a floor constitutes a greater and more direct hazard for the spread of fire from story to story than the localized passage of heat, flame, or hot gases through the floor construction. Avoidance of collapse is therefore of major importance in preserving the fire-barrier functions of floor constructions. In the following discussion of the significance of the thermal properties of the slabs with particular reference to reinforced concrete floors, emphasis is placed on structural stability rather than on heat transmission to the unexposed face.

It will be shown later that the ultimate strength of a reinforced concrete beam or slab depends in general on the yield strength of the reinforcing steel. Hence the load-carrying ability of a reinforced concrete floor construction can be expected to be closely related to the effect of high temperatures on the yield strength of the steel. If the yield strength is lowered by rise in temperature, extreme distress or structural collapse can be expected when a "critical" temperature is reached at which the yield strength becomes equal to the stress imposed on the steel.

These considerations provide a basis for estimating the probable period during which a reinforced concrete floor can carry its design load and for determining the thickness of concrete required to protect the steel and prevent collapse of floor slabs for given durations of exposure to fire. In order to make such determinations it is necessary to know the effect of temperature on the yield strength of steel, and the approximate time for critical temperatures to be reached at various distances from the exposed face during standard fire exposure. General information on the effect of temperature on yield strength, assembled from various sources, is presented in Figs. 8 to 11. Information on the time to attain critical temperatures was obtained from the data on temperature rise in the interior of the test slabs at various distances from the exposed face.

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[8] For a full rating, the specifications require that a duplicate floor specimen be subjected to a fire-exposure test for a period equal to one half of that indicated by the fire-endurance period of the first specimen (but not more than 1 hr.), and that immediately after this exposure the specimen must withstand the impact, eroding and cooling effects of a hose stream applied to all parts of the exposed face. This specimen must carry its design live load during the fire and hose stream tests. After cooling, but within 72 hr. after the hose stream application, it must also sustain a superimposed load equal to twice the design live load. The requirements of these auxiliary tests are omitted from the discussion in this paper because they are considered by the author to be less severe and of less moment than those of the Standard Fire Endurance Test.

Yield Strength the Significant Tensile Property of Reinforcing Steel:

It has long been known that the ultimate flexural strength of reinforced concrete beams and slabs, with steel percentages near the maximum for balanced reinforcement, is closely related to the "yield point" or "yield strength" of the steel. As yield stresses are developed in the steel the rate of elongation increases, and this causes the neutral axis to move closer to the compression surface of the flexural member. As a result the area in compression decreases until the concrete crushes.[9] Therefore "yield strength" becomes the significant tensile property of reinforcing steel. The term implies an increased rate of strain with respect to load. With ductile reinforcing steels having well-defined yield points (the stress at which there occurs a marked increase in strain without increase in stress) the limiting strength is clearly indicated by the "drop of beam" during tensile strength tests. With less ductile steels the yield strength may be based either on the values of permanent set or of total strain which are considered to mark the end of the structural usefulness or "structural yield point"[10] of the reinforced assembly. Some investigators [Johnston and Cox (**8**); Steinman (**11**)] have used yield strengths based on permanent sets of 0.2 per cent determined by the A.S.T.M. offset method[11] whereas others [Whitney (**12**); Mensch (**13**); Saliger (**14**)] have considered the yield strength of reinforcing steel in flexural members as the stress producing a strain of 0.004 or 0.005 in. per inch. Whitney (**9**) who has used a strain of 0.004 in. per in. (0.4 per cent) in defining a yield point stress states:

> "It is the total strain in the steel which causes disruption, not just the plastic strain which is considered in the determination of the yield point by the A.S.T.M. offset method. It would therefore probably be better to define the yield point as the stress producing a definite total strain. Additional tests are needed to show whether 0.004 is the best value."

The preceding remarks, which are based on the performance of reinforced concrete beams and slabs at normal temperatures, apply only to tests in which ultimate strengths were attained without prior failure in diagonal tension or bond. It should also be understood that in conventional design practice the allowable stress is usually about 50 per cent (range 45 to 55 per cent) of the yield strength of the reinforcing steel. Hence the ultimate load is equal to twice the dead plus live load contemplated in the design and will be attained by applying a superimposed load equal to one dead load plus 2 live loads. Consequently, if the dead load of a floor slab is equal to its design live load (an average case) the construction can carry up to 3 live loads before the steel is stressed to its yield point.

PUBLISHED DATA ON EFFECT OF ELEVATED TEMPERATURES ON STRENGTH OF CARBON STEEL

In considering the performance of concrete beams and slabs during fire exposure it is of major importance to have a clear picture of the effect of high temperatures on the strength properties of steel—particularly on the yield strength of the steel. Since there seems to be no published information on the properties of any of the various types and grades of commercial reinforcing bars at high temperatures, data for carbon steels, assembled from various sources, are presented to indicate approximately what is involved.[12]

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[9] The general effect of yield point of steel on ultimate strength of beams and slabs was demonstrated in tests made in recent years with high yield strength steels by Lyse and Wernisch (Fig. 4, reference (10)) and Johnston and Cox (Figs. 12, 13, reference (8)).

[10] This term was used by Johnston and Cox (8) in reporting the results of their beam tests. It represents loads about 10 per cent lower than the ultimate carried by the beams.

[11] A.S.T.M. Standard Methods of Tension Testing of Metallic Materials, (E 8 - 42) 1942 Book of A.S.T.M. Standards, Part I, p. 898.

Effect of Temperature on Stress-Strain Curves for Carbon Steel:

Figure 8 indicates stress-strain curves for tension tests of two carbon steels at various temperatures. The carbon contents, 0.37 and 0.48 per cent, are near the upper limits of carbon content of commercial reinforcing bars of intermediate and hard grade, respectively. These curves illustrate the effects of temperature rather than the effects of carbon content on the stress-strain characteristics of the steel. The tensile strength rises with increase in temperature up to about 500 to 600 F. and then decreases rapidly with further rise in temperature. Note that the sharp breaks in the stress-strain curves for temperatures of 65, 212, and 392 F. are absent in the curves for 572 F. and higher temperatures. The stresses at the breaks in the curves in the upper diagram indicate an increase in the "drop-of-beam" yield point with temperature up to at least 392 F. whereas in the lower diagram they show a decrease. However, regardless of whether the yield point is increased or decreased[13] by a moderate temperature rise, with further rise there ultimately occurs a marked reduction in yield strength which at 1000 F. averages about 50 per cent of the values at 70 F. There is also a marked reduction in the modulus of elasticity at the higher temperatures.

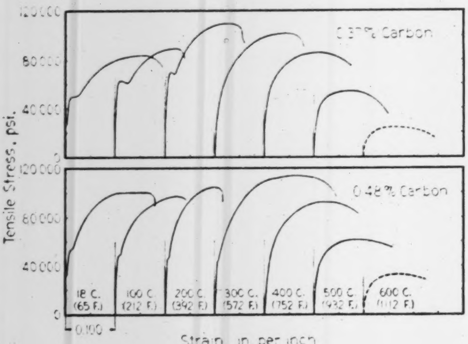

FIG. 8.—Stress-Strain Curves for Tension Tests of Two Carbon Steels at Various Temperatures.

Data from G. Welter (6).

Upper and lower diagrams taken from autographic records in Figs. 38 and 61, respectively, of reference (6) except the dash-line curves for 600 C. which were estimated from the ultimate strength relationship given in Figs. 37 and 60, respectively, from the reference cited.

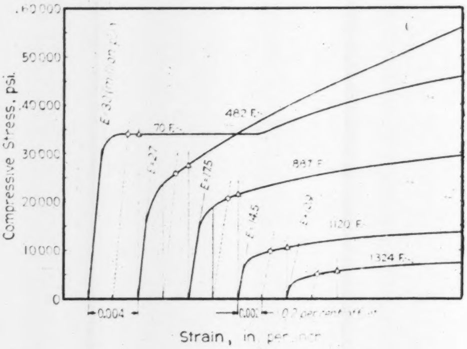

FIG. 9.—Stress-Strain Curves for Compression Tests of 0.20 per cent Carbon Steel at Various Temperatures.

Data taken from Fig. 11 and Table 6 of reference (2). The open circles on each curve represent yield strength values as determined by the offset method (A.S.T.M. Standard Methods of Tension Testing of Metallic Materials (E 8 - 42), 1942 Book of A.S.T.M. Standards, Part I, p. 898) using offset of 0.2 per cent. The open triangles represent the stress at a unit strain of 0.004 (0.4 per cent).

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[12] The general status of knowledge in 1937 on the strength of carbon steel at elevated temperatures is indidicated by the following statement by Frank T. Sisco, Editor of "The Alloys of Iron and Carbon" (reference (5)) taken from his summary of Chapter XII on Short-Time Elevated-Temperature Tests:

"Within the last decade many data on the effect of temperature on mechanical properties of ferrous alloys have been reported. Carbon steel loses strength so rapidly at elevated temperatures that alloy steels and special heat-resisting alloys are used if service conditions are severe; hence, most of these data were obtained on alloy steels rather than on carbon steels. As a result, despite the large amount of work which has been done, many fundamental facts about the high- and low-temperature properties of carbon steels cannot yet be stated definitely."

[13] It appears that the increase in yield point at the lower steps of temperature rise is likely to occur with steel made by the Bessemer process, whereas the decrease in yield point is a general characteristic of steel made by the open-hearth process. At the present time (May, 1943) over 95 per cent of the steel used to reinforce concrete (both new billet and rail steel) is made by the open-hearth process.

The general effect of temperature on the stress-strain curves for compression tests is about the same as for tension tests. This is evident from the curves of Fig. 9 for compression tests of a 0.20 per cent carbon steel which show the changes in shape and slope of the significant portions of the stress-strain relations for temperatures up to 1324 F. As in the case of the curves in Fig. 8 the sharp break at yield point in the curve for 70 F. is absent in the curves for higher temperatures. The modulus of elasticity decreased from the usual value

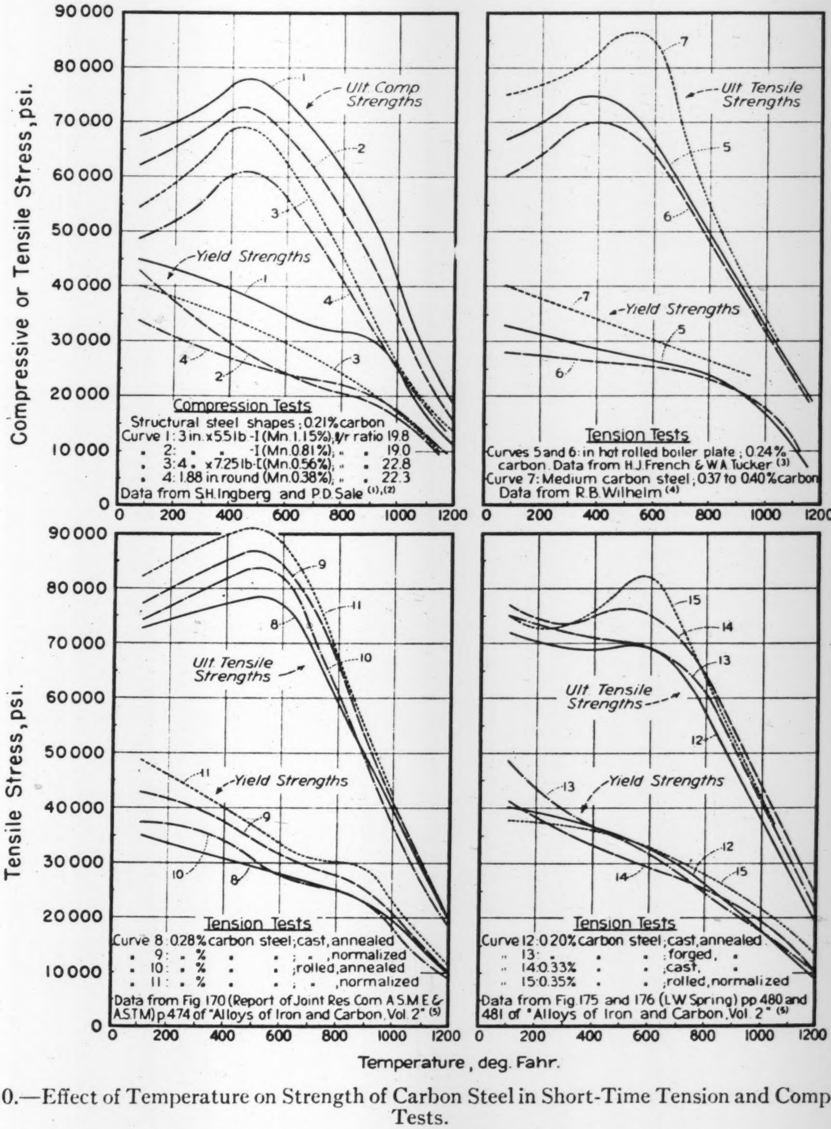

FIG. 10.—Effect of Temperature on Strength of Carbon Steel in Short-Time Tension and Compression Tests.

All yield strengths shown were determined by offset method (A.S.T.M. Standard Methods of Tension Testing of Metallic Materials (E 8 – 42)) using offset of 0.2 per cent.

of 30,000,000 psi. at normal temperatures to 10,900,000 in the test at 1324 F.

Comparison of Yield Strengths at High Temperatures by 0.2 per cent Offset and 0.4 per cent Strain Methods:

The curves of Fig. 9 are useful in showing that it makes little practical difference whether the yield strength of steel at high temperatures is based on the stress at a permanent set of 0.2 per cent (determined by the A.S.T.M. offset method) or by the stress producing a total strain of 0.004 in. per inch (0.4 per cent). In view of this, Fig. 11 was plotted by using data from various sources based on 0.2 per cent offset whereas corresponding data based on unit strains of 0.004 or other values could not be obtained since stress-strain relations were not available.

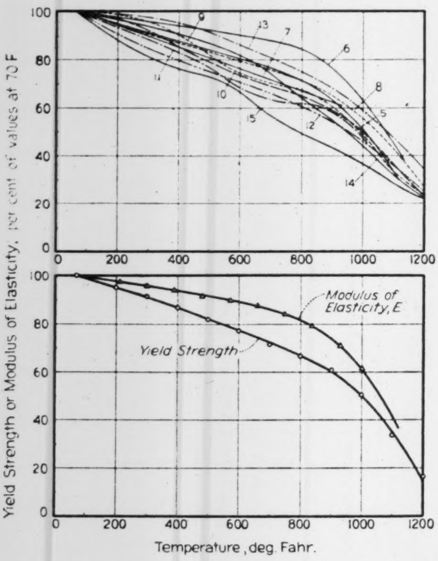

FIG. 11.—Effect of Temperature on Yield Strength and Modulus of Elasticity of Carbon Steel in Tension.

Curves in upper diagram were based on stresses given in the three diagrams of Fig. 10 for tension tests.

In the lower diagram the curve for yield strength is based on the average of all of the data in the upper diagram. The curve for modulus of elasticity is reproduced from Fig. 147, p. 439 "Alloys of Iron and Carbon," Vol. 2 (5) and is based on the averages for carbon steels as reported by H. J. Tapsell.

Effect of Temperature on Yield Strength and Modulus of Elasticity of Carbon Steel:

Figure 10 shows the effect of temperature on the strength in tension and compression of a variety of carbon steels. Although these curves represent variations in carbon content, heat treatment, casting, rolling, forging, and other manipulations they all indicate a very marked reduction in yield strength at 1000 F. This is clearly seen from the group of curves in the upper diagram of Fig. 11 which express the yield strengths in tension at various temperatures obtained from the curves numbered 5 to 15 in Fig. 10, in percentage of the corresponding yield strengths at 70 F. The average yield strength for this group of 11 curves is represented by the curve in the lower diagram of Fig. 11. Included in this diagram is a similar curve representing the average modulus of elasticity of carbon steels as reported by H. J. Tapsell. The latter agrees very well with such values of modulus of elasticity as are available for the tension and compression tests represented in Figs. 8, 9, and 10.

1000 F. the Critical Temperature for Reinforcing Steel in Flexural Members:

The yield strength curve in the lower diagram of Fig. 11 provides a basis for determining the critical temperature for reinforcing steel in concrete slabs or beams exposed to fire. The term *critical* temperature is used to denote the temperature in the reinforcing steel at which the flexural member is in distress and in danger of structural collapse. For all practical purposes it is the temperature at which the yield strength of the steel becomes equal to the steel stress developed by the dead and live loads.

In the average case, where the design

is based on a working stress of one half of the yield strength at normal temperatures, the yield strength curve of Fig. 11 indicates that at about 1000 F. the yield strength has been lowered to about 50 per cent of that at room temperature. Thus in the average case, 1000 F. is the critical temperature for reinforcing steel at which structural failure of flexural members can be expected unless the fire effects have so weakened the member in diagonal tension, bond, or compressive strength as to cause earlier failure. The value of 1000 F. was used in Fig. 12 and later diagrams as a basis for discussing the practical significance of the temperature data obtained in the slab tests with respect to the probable performance of reinforced concrete floor specimens.[14] When the design working stress in the steel is lower or higher than half that of the yield strength at normal temperatures, which was assumed for the average case, the critical temperature will be higher or lower than 1000 F.

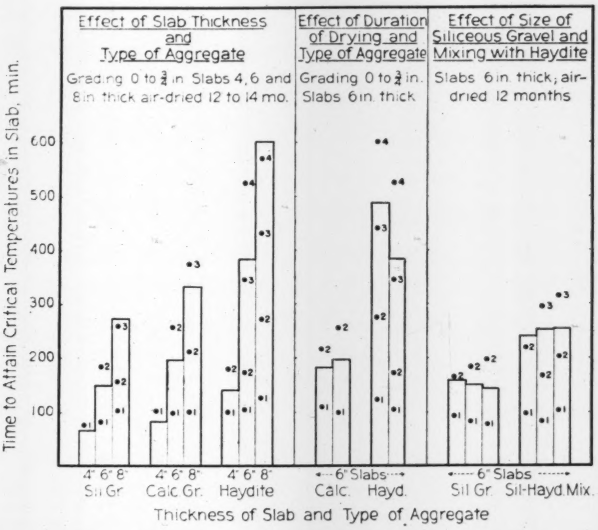

FIG. 12.—Comparison Between Time to Attain 1000 F. at Various Distances from Exposed Face of Slab with Time to Attain Average Rise of 250 F. on Unexposed Face of Slab.

Data from Table I (columns 8, and 20–23)

Heights of columns represent the time to attain an average rise of 250 F. on the unexposed face of slab. This time represents the fire-endurance period of the slab provided it meets the other requirements of the standard fire test specification as a structural member.

The height of each plotted point indicates the time in minutes and the accompanying numeral the number of inches from the exposed face of the slab when an average temperature of 1000 F. was indicated by two thermocouples embedded in the upper and lower halves of the slab. (Locations for each pair of thermocouples at given distances from exposed face are shown in Fig. 1).

TEMPERATURE RISE RELATIONSHIPS FOR SLAB TESTS AND THEIR SIGNIFICANCE

The data of Figs. 12, 13, and 14 summarize the thermal properties of the test slabs and illustrate how the results can be applied in the design of floor slabs of substantially uniform thickness

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[14] Although the modulus of elasticity has been reduced to 60 per cent of normal at 1000 F. (lower diagram Fig. 11) it will be apparent from a study of the stress-strain curves in Fig. 9 that the determination of yield strength either by the 0.2 per cent offset method or 0.4 per cent limiting strain method automatically includes the increased strain resulting from a change in the slope of the stress-strain curve. The reduction in modulus of elasticity with increase in temperature of the steel is important, however, because it results in an increase in the deflection of a flexural member in proportion to the increase in strain of the steel although the stress remains constant. This increase of deflection together with the deflection or bowing toward the fire, which results from differential expansion, can be expected to affect the sequence of events which contribute to ultimate failure under design load.

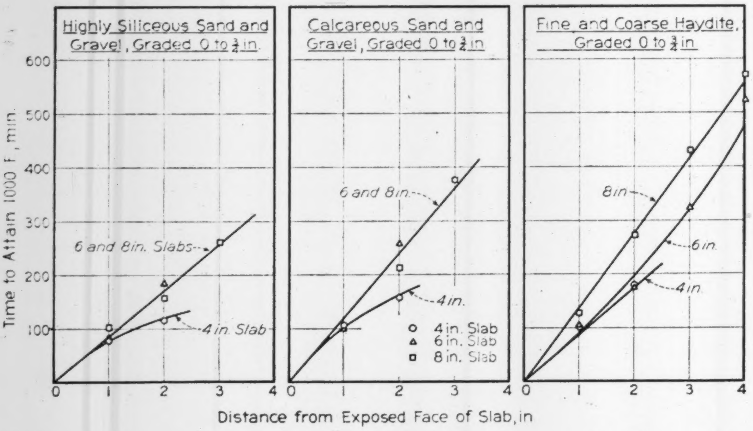


FIG. 13.—Relationships Between Distance from Exposed Face and Time to Attain 1000 F. in Slabs of Different Thickness and Types of Aggregates.

Data from Table I (Columns 20–23.)

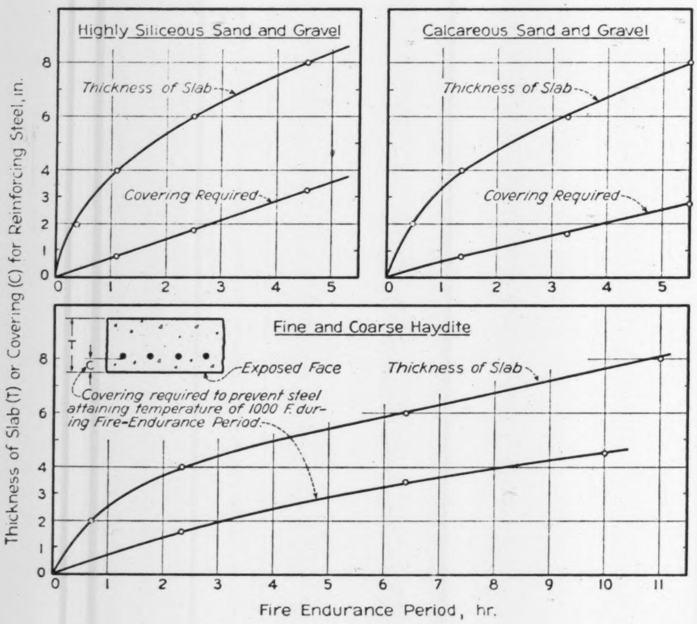


FIG. 14.—Relation between Fire-Endurance Period, Thickness of Slab and Covering for Steel for Different Aggregates.

The relation between fire-endurance period and thickness is the same as indicated in the right diagram of Fig. 4. It is based on the data in columns 4 and 8 of Table I for the attainment of an average rise of 250 F. as indicated by thermocouples on the unexposed face of the slab.

The plotted values of covering required for slabs of different thickness were taken from the graphs of Fig. 13.

so that they will perform approximately as intended for given periods of standard fire exposure.

Comparison of Time to Attain 1000 F. within Slab with Potential Fire Endurance Period:

The height of the plotted points in the diagrams of Fig. 12 give a general picture of the time to attain 1000 F. at various distances from the exposed face of each slab. This time represents the approximate period during which reinforcing steel placed at these same distances from the exposed face of a floor slab of similar makeup would be protected from attaining the critical temperature of 1000 F. during standard fire exposure. In other words each plotted point represents the approximate period during which a given slab with the indicated covering for the steel could be expected to carry its normal design load if it were tested as a floor slab with exposure to fire from the under side. The heights of the columns in Fig. 12 indicate the potential fire endurance period which can be expected to be realized in such floor slab specimens (based on time to attain an average rise of 250 F. on the unexposed face) provided the reinforcing steel is protected sufficiently to avoid high steel temperatures, and thus structural collapse under the design load, during this period.

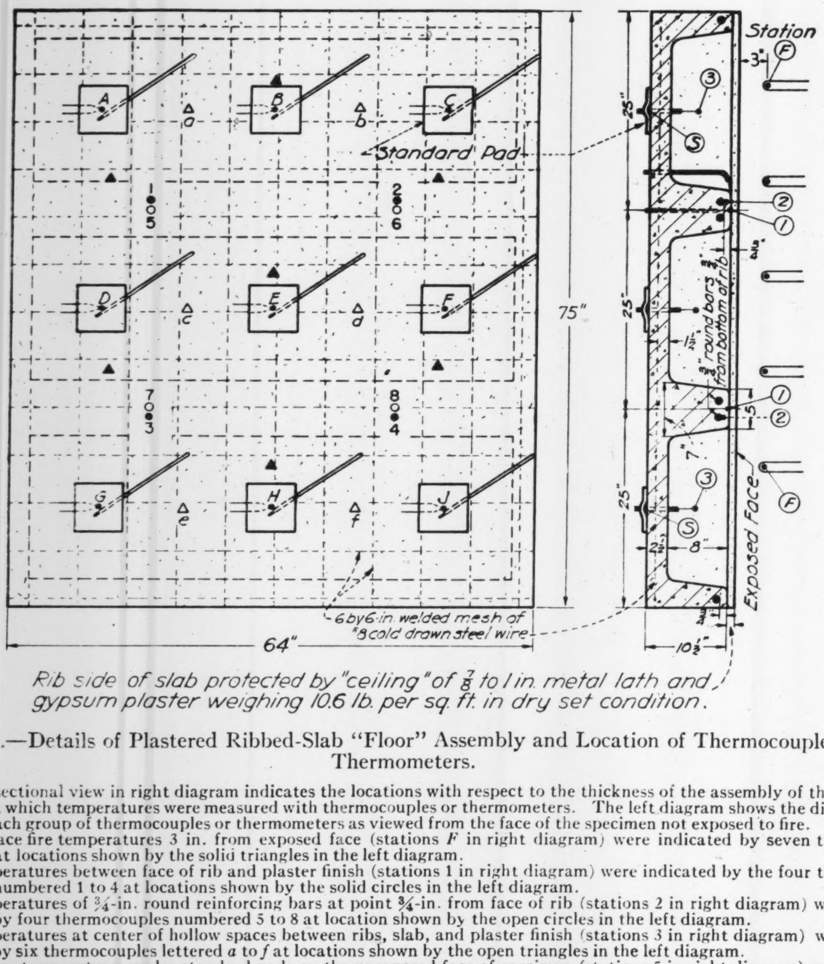
Relationships Between Distance from Exposed Face and Time to Attain 1000 F.:

The relationships shown in the diagrams of Fig. 13 are believed to give a reliable indication of the approximate time at which steel placed at a given distance from the exposed face of solid floor slabs of similar makeup will attain a temperature of 1000 F. during standard fire exposure. These relationships are influenced by a variety of factors associated with a given slab such as thickness, mass, moisture content, type of aggregate, differential fire effects near exposed face (shattering, decarbonization), etc. These factors account for the fact that the relationship indicated by the data for the 4-in. slabs of the two natural aggregates is curvilinear instead of rectilinear as was obtained for the 6- and 8-in. slabs with the same natural aggregates. They also account for the separate relationships for the 4-, 6-, and 8-in. slabs made with the manufactured light-weight Haydite aggregate. In general the lines through the plotted points serve to minimize any inconsistencies in individual values although each of these represents the average indication of two thermocouples.

Relationship Between Fire Endurance Period, Thickness of Slab, and Thickness of Covering for Steel for Different Aggregates:

The upper curve in each diagram of Fig. 14 indicates the approximate thickness of slab required for various fire endurance periods. The lower curve indicates the approximate thickness of the concrete covering required to prevent the steel from attaining the critical temperature of 1000 F. during the fire-endurance period. If it is desired that a floor slab have a fire-endurance period of 3 hr. the curves indicate that with highly siliceous sand and gravel the slab should be about 6.5 in. thick and the covering for the reinforcing steel should be about 2.2 in. For calcareous sand and gravel the slab thickness should be about 5.7 in. and the steel covering about 1.6 in. For fine and coarse Haydite the slab thickness should be 4.4 in. and the steel covering about 2.0 in.

In the case of Haydite the covering required to protect the steel from critical temperatures is high in proportion to the over-all thickness of the slab required to prevent the attainment of critical temperatures (average rise of 250 F.) on the unexposed face or top of the floor slab. A similar but less pronounced result is evident with the highly siliceous natural aggregate at the higher fire-endurance periods. In designing floor slabs for a specific fire-endurance period the first step is *to provide the necessary cover for the steel* and then to determine the thickness of the slab above the steel from the depth (or *jd*) needed to satisfy the normal requirements for strength and economy. The over-all thickness of the slab thus obtained should then be checked to see that it is not less than required by the curves of Fig. 14 for the type of aggregate to be used.



Rib side of slab protected by "ceiling" of ⅞ to 1 in. metal lath and gypsum plaster weighing 10.6 lb. per sq. ft. in dry set condition.

FIG. 15.—Details of Plastered Ribbed-Slab "Floor" Assembly and Location of Thermocouples and Thermometers.

The sectional view in right diagram indicates the locations with respect to the thickness of the assembly of the "stations" at which temperatures were measured with thermocouples or thermometers. The left diagram shows the distribution of each group of thermocouples or thermometers as viewed from the face of the specimen not exposed to fire.

Furnace fire temperatures 3 in. from exposed face (stations *F* in right diagram) were indicated by seven thermocouples at locations shown by the solid triangles in the left diagram.

Temperatures between face of rib and plaster finish (stations 1 in right diagram) were indicated by the four thermocouples numbered 1 to 4 at locations shown by the solid circles in the left diagram.

Temperatures of ¾-in. round reinforcing bars at point ¾-in. from face of rib (stations 2 in right diagram) were indicated by four thermocouples numbered 5 to 8 at location shown by the open circles in the left diagram.

Temperatures at center of hollow spaces between ribs, slab, and plaster finish (stations 3 in right diagram) were indicated by six thermocouples lettered *a* to *f* at locations shown by the open triangles in the left diagram.

Surface temperatures under standard pads on the unexposed face of specimen (stations 5 in right diagram) were indicated by nine thermocouples and nine thermometers lettered *A*, *B*, *C* . . . *J* in the left diagram.

It should be pointed out that the preceding discussion has assumed that the concrete covering the steel remains in position for the full fire-endurance period. This may only be partially realized under actual fire exposures. Concrete made with aggregates which show the greatest expansions and disruptive effects can be expected to fall off more readily than concrete made with the more stable aggregates. The high siliceous aggregates fall in the former class whereas the calcareous aggregates and Haydite fall in the latter.[15] On the other hand, it may be stated that the loss of concrete covering will usually be local and may not extend to the steel so that the structural integrity of the entire slab will not be impaired until the average temperature of the steel over the entire area has attained critical values.

TABLE IV.—TESTS OF BARE AND PLASTERED RIBBED-SLAB SPECIMENS.

Concrete.—1:2:3½ mixture by loose volume of normal strength portland cement, calcareous sand and gravel (Elgin) graded 0 to No. 4 sieve and No. 4 sieve to 1-in., respectively. Plastic consistency 5 to 6-in. slump placed by hand rodding. Air-dry weight at time of test 149 lb. per cu. ft.

Curing.—Each ribbed-slab section was cured moist in mold for 28 days. Specimen F-1 was seasoned in air of laboratory for 56 days before fire-endurance test. Specimen F-2 was seasoned in air of laboratory for 28 days before plastering and 30 days after plastering or a total of 58 days before fire endurance test. Slabs cast July 22, 1930, and tested October 14 and 16, 1930.

Plastering.—Gypsum plaster was applied in three coats on diamond mesh ribbed metal lath weighing 3.0 lb. per sq. yd. The combined thickness of the three coats was ⅞ to 1-in. over the bottom of the ribs of the concrete slab. The weight per square foot of the dry-set materials in each coat was as follows: scratch coat 4.6 lb., brown coat 5.2 lb., finish coat 0.5 lb.—total plaster 10.3 lb. The gypsum and sand were proportioned on a dry-weight basis as follows: 1:2 for the scratch coat and 1:3 for the brown coat. The lath was securely held to the ribs of the slab by lath nail inserts which had been cast into the ribs. The head ends of these nails were firmly embedded in the concrete and the point ends protruded sufficiently so that they could grip the mesh of the lath when they were bent flat against the bottom of the concrete rib.

See Fig. 15 for location, distribution and number of thermocouples.

The fire-endurance period in column 8 is based upon the time at which nine No. 20 B. & S. gage calibrated iron-constantan wire thermocouples symmetrically distributed (See Fig. 14) and in contact with the unexposed face under standard pads indicated an average rise of 250 F. or a rise at any individual point of 325 F. For test No. F-1 the average and maximum rise was obtained at 46 and 45 min., respectively, and for test No. F-2 at 158 and 163 min., respectively. The values of fire endurance in column 9 were determined from the curve for solid slabs in Fig. 4 for calcareous gravel.

Values in parentheses are percentages of the total weight of assembly given in column 6.

Test	Specimen	Average Weight of Parts of Assembly, lb. per sq. ft. of slab			Weight of Assembly, lb. per sq. ft. of slab		Fire-Endurance Period, min.		Average Temperature of Steel in Ribs (Station 2, Figs. 15 and 16) at Time Indicated, deg. Fahr.			
		Slab (2½ in.)[a]	Rib (6 by 8 in.)[b]	Plaster (⅞ to 1 in.)[c]	Actual Total Amount	Amount Effective During Fire Exposure	By Actual Test	As Determined from Weight, Col. 7, and Weight Relation, Fig. 4, for Calcareous Gravel	1 hr.	1½ hr.	2 hr.	3 hr.
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
No. F-1	Bare	31.3 (55%)	25.5 (45%)	0.0	56.8	31.3 (55%)	45	40	1040	1230	...	...
No. F-2	Plastered	31.3 (46%)	25.5 (38%)	10.6 (16%)	67.4	67.4 (100%)	158	157	300	460	580	775

[a] Weight of air-dry concrete and welded mesh (31.0 plus 0.3 lb.).
[b] Weight of air-dry concrete and ¾-in. reinforcing bars (24.0 plus 1.5 lb.).
[c] Weight of metal lath, and dry set plaster (0.3 plus 10.3).

III. RESULTS OF TESTS OF BARE AND PLASTERED RIBBED-SLAB SPECIMENS AND THEIR SIGNIFICANCE

The tests of the two rib-type specimens provided information on the thermal properties of assemblies similar to unplastered and plastered ribbed-slab floors. Their significance was materially enhanced by the relationships developed by the tests on the solid slabs and *vice versa*. The discussion which follows brings out several points of practical interest, particularly regarding the manner in which plaster and metal lath may function to increase the fire resistance of floor constructions.

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[15] An excellent discussion on the influence of aggregate on the behavior of concrete during fire exposure will be found in a paper by S. H. Ingberg (7).

SPECIMENS AND TEST PROCEDURE

Figure 15 gives the general details of the $5\frac{1}{3}$ by $6\frac{1}{4}$-ft. specimen which simulated a section of a reinforced concrete ribbed-slab floor that was finished on the "ceiling" side of the ribs with metal lath and gypsum plaster. A companion specimen identical in every respect was tested without the plaster finish. Both specimens were cast horizontally of plastic concrete containing calcareous sand and gravel aggregate graded 0 to 1 in. They were tested vertically in the same manner as the solid slabs described previously. Each specimen was tested with the rib side toward the fire to simulate exposure to fire from the under side of a corresponding floor assembly. No load was imposed on these specimens either during or after fire exposure. Further details are given in the notes of Fig. 15 and of Table IV. The time-temperature curves are given in Fig. 16 and the significant results of the tests in Table IV.

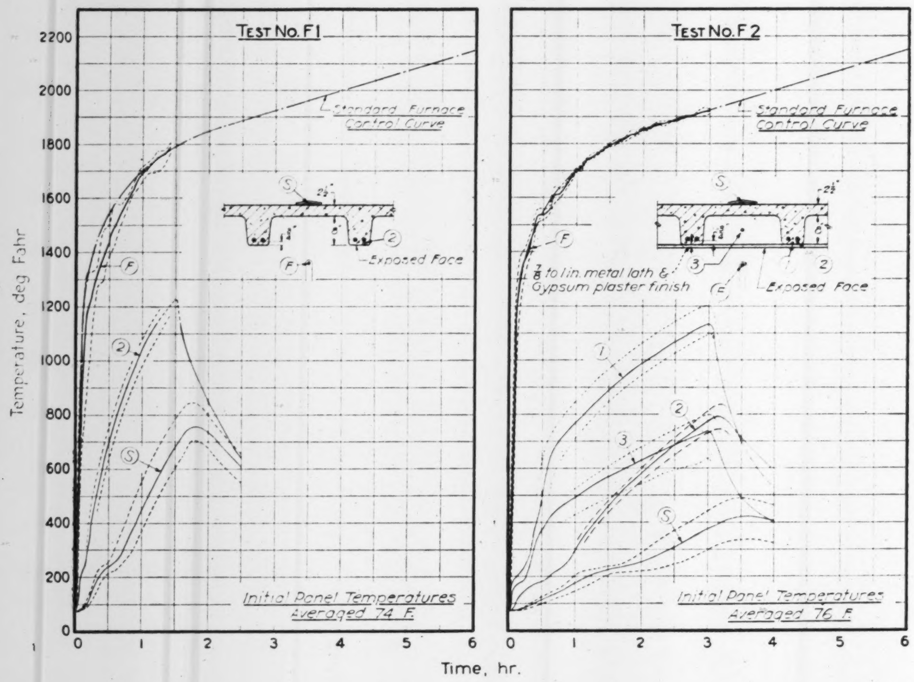

FIG. 16.—Time-Temperature Curves for Tests F-1 and F-2 of Bare and Plastered Ribbed-Slab Specimens.

DISCUSSION OF RESULTS

Effectiveness of Plaster:

Column 8 (Table IV) indicates that the bare specimen, F-1, attained one of the critical end-point temperatures (average rise of 250 F. or maximum rise of 325 F.) on the unexposed face at 45 min. whereas the plastered specimen, F-2, did not attain such temperatures until after 158 min. exposure. Hence, by adding about 10.6 lb. of lath and plaster, (increase in weight 18.7 per cent) a fire endurance period 3.5 times that of the bare specimen was obtained.

These results furnish a striking example of how an apparently minor element in an assembly may exert an important influence on its over-all performance during fire exposure. In this case the plaster functioned in two ways to retard the transmission of high temperatures to the unexposed face of the assembly: (1) by serving in its normal capacity as an insulating medium and barrier to the direct action of fire, (2) by bringing the potential heat-absorbing capacity of the concrete ribs into play. Moreover, it greatly retarded the rise in temperature of the reinforcing steel in the ribs of the specimen. These functions will become clear from a study of the cross-sections for tests F-1 and F-2 in Fig. 16 and the discussion in the following paragraphs.

Only Slab Portion of Unplastered Rib-Type Specimen Effective in Retarding Temperature Rise on Unexposed Face:

In the unplastered specimen the $2\frac{1}{2}$-in. slab was exposed to the direct action of the fire. Therefore the development of critical temperatures on the unexposed face was almost entirely a function of the thermal properties of a plain solid slab $2\frac{1}{2}$ in. thick, weighing 31.3 lb. per sq. ft. as indicated in column 3 of Table IV. This statement is based on the fact that the actual fire-endurance period of 45 min. (column 8) was only 5 min. more than the 40 min. (column 9) which would be expected for a simple slab of this weight (or thickness) from the relationships shown in Fig. 4 for calcareous gravel aggregate. Thus as indicated in column 7 of Table IV only 31.3 lb. or 55 per cent of the total weight of the unplastered specimen, F-1, was effective in retarding the development of high temperatures on the unexposed surface of the slab.

All Parts of Plastered Rib-Type Specimen Effective in Retarding Temperature Rise on Unexposed Face:

In the plastered specimen, F-2, the heat which passed through the plaster into the air space between the ribs was absorbed by the relatively cool mass of concrete in the ribs as well as in the slab. Since the rate of temperature rise in this air space was moderate (curve 3, test F-2, Fig. 15) it appears that the full heat-absorbing capacity of the mass of concrete in the ribs was brought into play. This mass had an average weight of 25.5 lb. per sq. ft. (column 4) and was equivalent to that of a solid slab 2.05 in. thick since the air-dry weight of the concrete was 149 lb. per cu. ft. On this basis the thermal properties of the assembly may be considered to be equivalent to that of a solid slab of concrete 4.55 in. thick (2.5 plus 2.05 in.) plus 0.94 in. of plaster ($\frac{7}{8}$-in. to 1-in.) or a total thickness of about 5.5 in. The effective weight of this slab would be the same as that of the actual plastered specimen or 67.4 lb. per sq. ft. as in columns 6 and 7 of Table IV. Its fire-endurance period on the basis of this weight and the weight relations in Fig. 4 for calcareous gravel could be expected to be about 157 min. as shown in column 9. Its fire-endurance period as a solid slab 5.5 in. thick can similarly be determined from the thickness relation in Fig. 4 for calcareous gravel. This likewise indicates a probable fire-endurance period of about 157 min. assuming that the thermal properties of gypsum plaster and concrete are about the same for the thickness involved. Therefore, in this case the actual fire-endurance period of 158 min. (column 8) was only 1 min. higher than the 157 min. which could be expected from the weight relations given in Fig. 4.

Approximate Potential Fire Endurance Period of Composite Assemblies May Be Estimated from Weight Relationships of Fig. 4:

While the foregoing close agreement may have been accidental, nevertheless it is believed that when interpreted with care and judgment the weight relationships given in Fig. 4 can be used to obtain reasonably accurate estimates of the relative fire-endurance periods of other composite assemblies. This belief is based on the similarity of the weight relationships for the solid slabs with those established for the dry-tamped concrete used in walls of hollow masonry units,[16] and on the fact that estimates based on the latter relationships have been verified by standard fire-endurance tests on large hollow concrete masonry walls when due allowance was made for the differences in testing technique involved.[17]

Effect of Plaster on Temperature Rise in Reinforcing Steel:

Values in columns 10 to 13 of Table IV (taken from curves for station 2, tests F-1 and F-2, Fig. 16) indicate the marked difference between the average temperature of the reinforcing steel in the ribs of the bare and plastered specimens. These results show that the addition of plaster delayed materially the attainment of critical temperatures in the steel and that a plastered construction such as that used in test F-2 will probably carry its design load at least three times longer during standard fire exposure than an unplastered construction such as that used in test F-1.

In the unplastered specimen of Test F-1 the steel attained the critical temperature of 1000 F. in 55 min. or 10 min. after its potential fire-endurance period of 45 min. (column 8, Table IV) had been determined by the temperature rise on its unexposed face. From this it appears that with a similar unplastered construction the fire-endurance period would probably be determined by temperature rise on the top face rather than by structural failure under the design load.

In the plastered specimen (test F-2) the average temperature of the steel in the ribs was only 300 F. at 1 hr. as compared with 1040 F. for the unplastered specimen (test F-1), and at 3 hr. it was 775 F. or about 225 F. below the critical temperature of 1000 F. for reinforcing steel. According to the curve in the lower diagram of Fig. 11 the yield strength at 775 F. could be expected to be about 64 per cent of the yield strength at 70 F. On this basis a similar plastered floor construction could be expected to carry its design load for at least 3 hr. although, as indicated by column 8 of Table IV, its fire-endurance period would probably actually be determined by the temperature rise on the unexposed top face at slightly over 2½ hr. (158 min.). In this, as well as in the preceding paragraph, it is assumed that the design is based on an allowable steel stress of one half of the yield strength of steel at normal temperatures.

Temperature Rise of Steel in Rib-Type Specimen Influenced by Various Factors:

The data on temperatures developed in the steel in the ribs of both the bare and plastered specimens illustrate several other points worthy of mention. For instance, the tests of specimen F-1 show that the temperature rise of steel embedded in concrete ribs or similar narrow sections will be more rapid than indicated by the relationships in Fig. 13 which are based on tests of solid slabs. This is not surprising since the cross-sectional detail of this specimen in Fig. 16 shows that the sides as well as the bottom of the ribs were directly exposed to fire. Curve 2 (Fig. 16) shows that a temperature of 1000 F. was actually attained $\frac{3}{4}$-in. from the bottom of the rib (station 2) at about 55 min. whereas the relationships of Fig. 13 indicate that 1000 F. would not be expected in solid slabs of similar concrete (calcareous sand and gravel) at a distance of $\frac{3}{4}$-in. from the exposed face for at least 80 min.

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[16] See Fig. 8 of reference (17).

[17] This was pointed out in the last footnote on page 10 of reference (17) as follows:

Although the particular test technique followed gives a reliable basis for comparison of the relative fire-resistant properties of the various wall assembles, it is of interest to point out that the fire-endurance periods obtained in this investigation with 5½ by 6-ft. walls are approximately 10 per cent lower than the periods obtained in standard tests of 10 by 11-ft. walls at Underwriters' Laboratories which were constructed of units identical in design and composition. The fire-endurance periods derived in this investigation may, therefore, be considered as conservative criteria of the performance of standard size wall specimens. For further information on this point see Article 8A, Appendix A.

In the case of the plastered specimen (F-2, Fig. 16) curves 3 and 1 indicate that the sides of the ribs were exposed to much lower temperatures than the bottom of the ribs. The temperature of the steel at station 2 (curve 2) was therefore mainly determined by the flow of heat through the combined thickness of the plaster and concrete covering over the steel at the bottom of the rib. This covering was about 1.7 in. thick (0.94 in. plaster plus 0.75 in. concrete) and from the trend of curve 2 would have prevented attainment of 1000 F. in the steel for about 4.2 hr. (250 min.) whereas according to the relationships in Fig. 13 the same thickness of solid concrete made with calcareous gravel could not be expected to prevent the steel from reaching 1000 F. for more than about 210 min. or 3.5 hr. From these same relationships it appears that about 2.1 in. of solid concrete would produce the same effect as the 1.7 in. of plaster and concrete.

Although the heat-insulating value of the gypsum plaster was somewhat greater than that of the solid concrete made with calcareous aggregate (compare curve 1, Test F-2, Fig. 16, with curves 1 in. from the exposed face of solid slabs 4, 6, and 8 in. thick in tests No. 119, 116, and 115 in Figs. 19 and 20) the difference was not sufficient to explain the over-all effectiveness of the layer of plaster plus the layer of concrete. It is believed that the greater effectiveness of the plaster and concrete combination was mainly the result of an interruption in heat flow at the boundary between the plaster and the concrete.

Because of the interference offered by the mesh of the metal lath the total area of contact between the plaster keys and the bottom of the ribs was inherently lower than with direct application of plaster to the concrete. Early during fire exposure, most, if not all, of this contact was broken by the expansion of the plaster and the metal lath. This was followed by other differential movements between the layer of plaster and the concrete particularly with the large shrinkage which occurs when gypsum dehydrates at elevated temperatures, whereas the concrete continues to expand as the temperature increases. This shrinkage also reduced the thickness of the plaster and increased the degree of separation between the two materials.

It will be apparent that even a small air gap (formed in whatever manner) would interrupt the direct flow of heat by conduction from plaster to concrete. Moreover, even if the contact between plaster and concrete were perfect throughout the fire-exposure period the flow of heat across the bonded joint could not be expected to be as high as through the numerous pieces of $\frac{1}{4}$ to 1-in. aggregate[18] which are so situated as to provide direct thermal (as well as structural) connecting links between the various parts of the heterogeneous mass of concrete.[19]

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[18] It should be understood that the conductivity of the common dense aggregate is higher than that of the more porous mortar which binds them together. The effect of grading aggregate is discussed in pages 70 to 72, reference (17).

From the preceding discussion it will be evident that the temperature rise of steel in the rib-type specimens was appreciably *more* rapid in the bare specimen and appreciably *less* rapid in the plastered specimen than in solid slabs. Therefore it would be incorrect to use the relationships in Fig. 13, based on tests of solid slabs, to determine the temperature of steel in narrow sections such as ribs or beams or in composite assemblies.

IV. CONCLUSIONS

The data presented in this paper seem to justify the following conclusions:

1. During exposure to fire the expansion, deflection, temperature transmission and general behavior of solid slabs cast from plastic concrete varies mainly with the type of aggregate used. With mixtures of equal volumes of two widely different aggregates the thermal properties of the concrete are practically intermediate between those of concrete made from each of the aggregates separately.

2. The fire-endurance period of solid concrete slabs increased greatly with increase in thickness. For each type of aggregate the fire-endurance period was in general doubled when the slab thickness was increased 35 to 40 per cent.

The relationships between fire-endurance period and the air-dry weight per square foot of the solid concrete slabs is similar to the relationships developed in tests of walls of hollow concrete masonry units where it was found that the fire-endurance period increased as an exponential function of the average air-dry weight per square foot of wall assembly.

Comparison of these relationships indicates that pound for pound the relatively dense concrete in the solid slabs was from 15 to 30 per cent less effective in retarding temperature rise on the unexposed face in the case of the two natural aggregates (siliceous and calcareous gravel) than the corresponding dry-tamped concrete in hollow masonry walls. The Haydite concrete was from 10 to 40 per cent more effective in the solid slabs than in the walls of hollow masonry units. The greater effectiveness of the Haydite concrete in the solid slabs is mainly due to tenaciously-held moisture within the thicker Haydite slabs.

3. In these tests the fire-endurance period of solid concrete bearing walls was determined by the temperature rise on the unexposed face and not by structural failure under working load (see conclusion 10).

4. Solid concrete bearing walls 6 and 8 in. thick and of normal story height (10 to 11 ft.) may be expected to remain stable under uniformly distributed working loads of at least 400 to 500 psi. during and after severe fire exposure. This statement applies only to dense walls of concrete unrestrained at the ends, and containing not less than 5.2 sacks (490 lb.) of portland cement per cubic yard. The aggregate may vary widely as to type (siliceous, calcareous, Haydite) and size range (0 to $\frac{3}{8}$, 0 to $\frac{3}{4}$, or 0 to $1\frac{1}{2}$ in.) but should preferably be well graded.

5. Study of the allowable steel stresses used in conventional design practice and of published data on the general effect of elevated temperatures on the yield

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[19] The effect of eliminating the thermal connecting links of aggregate in concrete as well as the possible effect of small air gaps was indicated by the results of test No. 161A (Table 26, page 124, reference (17)). In this test the units were of the "duplex" type made by cementing two 4 by 8 by 16-in. 3-core tile units together by a solid joint ⅛-in. thick between adjacent faces. The test indicated that for the weight of concrete involved, the fire-endurance period was 25 per cent higher than with walls of the single-unit type in which the continuity of the concrete was not interrupted by joints.

strength and elastic properties of carbon steels indicates that 1000 F. is the critical temperature for carbon steel reinforcement in flexural concrete members.

6. The fire-endurance period of solid reinforced concrete floor slabs may be expected to be closely related to the approximate time to attain critical temperatures in the reinforcing steel nearest the exposed face. The thickness of concrete covering necessary to provide protection to the reinforcing steel to prevent distress or structural failure of solid floor slabs for given fire-endurance periods, was established for the aggregates used in the tests (Fig. 14).

7. In ribbed-slab floor construction the attainment of critical temperatures on the unexposed face and in the steel reinforcement of the ribs can be greatly retarded by using a ceiling of metal lath and gypsum plaster.

8. When interpreted with care and judgment the weight relationships established for solid slabs can be used to estimate the relative potential fire-endurance periods (temperature rise on unexposed face) of composite assemblies.

9. The test data for solid slabs should not be used to estimate the temperature rise of steel in narrow sections such as ribs or beams in composite assemblies.

10. In extending the results of these tests on solid concrete slabs $5\frac{1}{3}$ ft. wide and 6 ft. high to larger slabs in walls or floors, consideration should be given to the effect of those factors or combinations of factors which may influence the behavior of large slabs in actual building construction. Height of wall slab, degree of restraint, distribution and character of superimposed load, type of aggregate, intensity and extent of fire exposure, and amount of steel reinforcement are some of the factors which may influence the stability and integrity of large wall or floor slabs when exposed to severe fire.

Acknowledgment:

The author wishes to acknowledge the assistance of W. R. Johnson and W. A. Ebert of Portland Cement Association Research Laboratory in the conduct of the tests and in the preparation and plotting of the data for the time-temperature curves.

APPENDIX

COMMENTS ON VALUES OF FIRE-ENDURANCE PERIOD

The fire tests of the hollow concrete masonry walls in the first investigation and of the solid and rib-type slabs in this investigation were made to develop precise information on the effect of various factors in materials and construction and not to determine specific fire-resistant ratings. In order to develop with a high degree of precision the information desired, a more exacting test procedure was followed than that required by the standard fire test specifications. This procedure gave fire-endurance periods about 10 per cent lower for the masonry walls and at least 8 per cent lower for the slab specimens than would be obtained in standard classification tests of 10 by 10 ft. walls under the requirements of the current A.S.T.M. specifications. The 8 per cent reduction in the case of the solid slab tests is the result of the combined effect of the variations from standard procedure described in (*a*) and (*b*) below:

(*a*) The fire-endurance periods are based on the indications of No. 20 B. & S. gage thermocouples (wire 0.032 in. in diameter) which were somewhat finer, and hence more sensitive to temperature changes than the No. 18 B. & S. gage thermocouples (wire 0.040 in. in diameter) or the thermometers (stem not over 5/16-in. in diameter) permitted under the specifications. With the

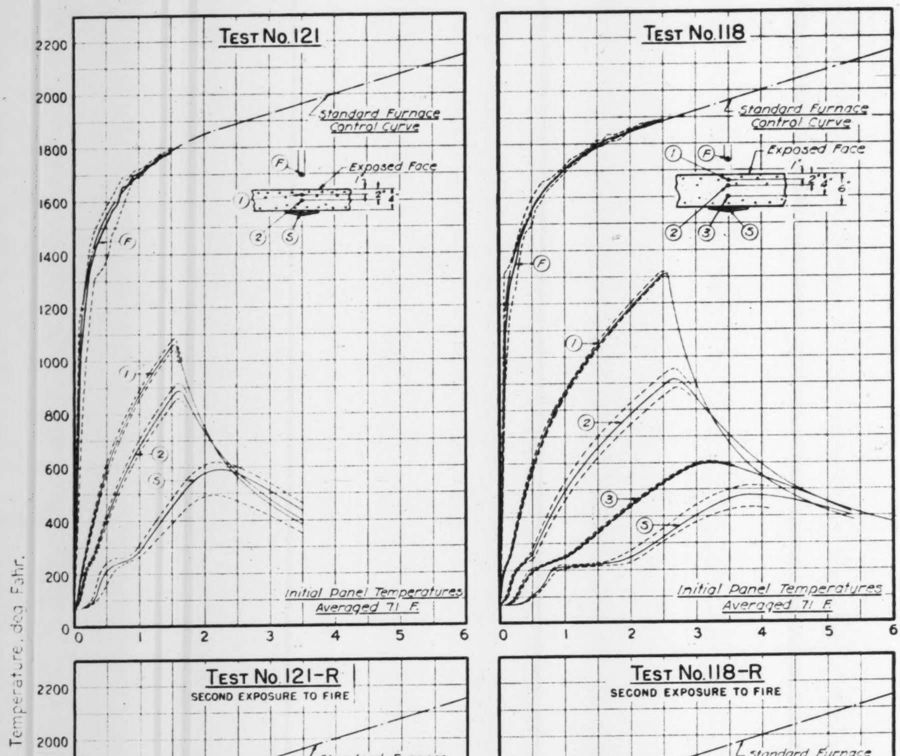

FIG. 17.—Time-Temperature Curves for Tests Nos. 121, 121-R, 118, and 118-R.

4- and 6-in. slabs; siliceous gravel, graded 0 to ¾-in.; air-dried 12 to 14 months.

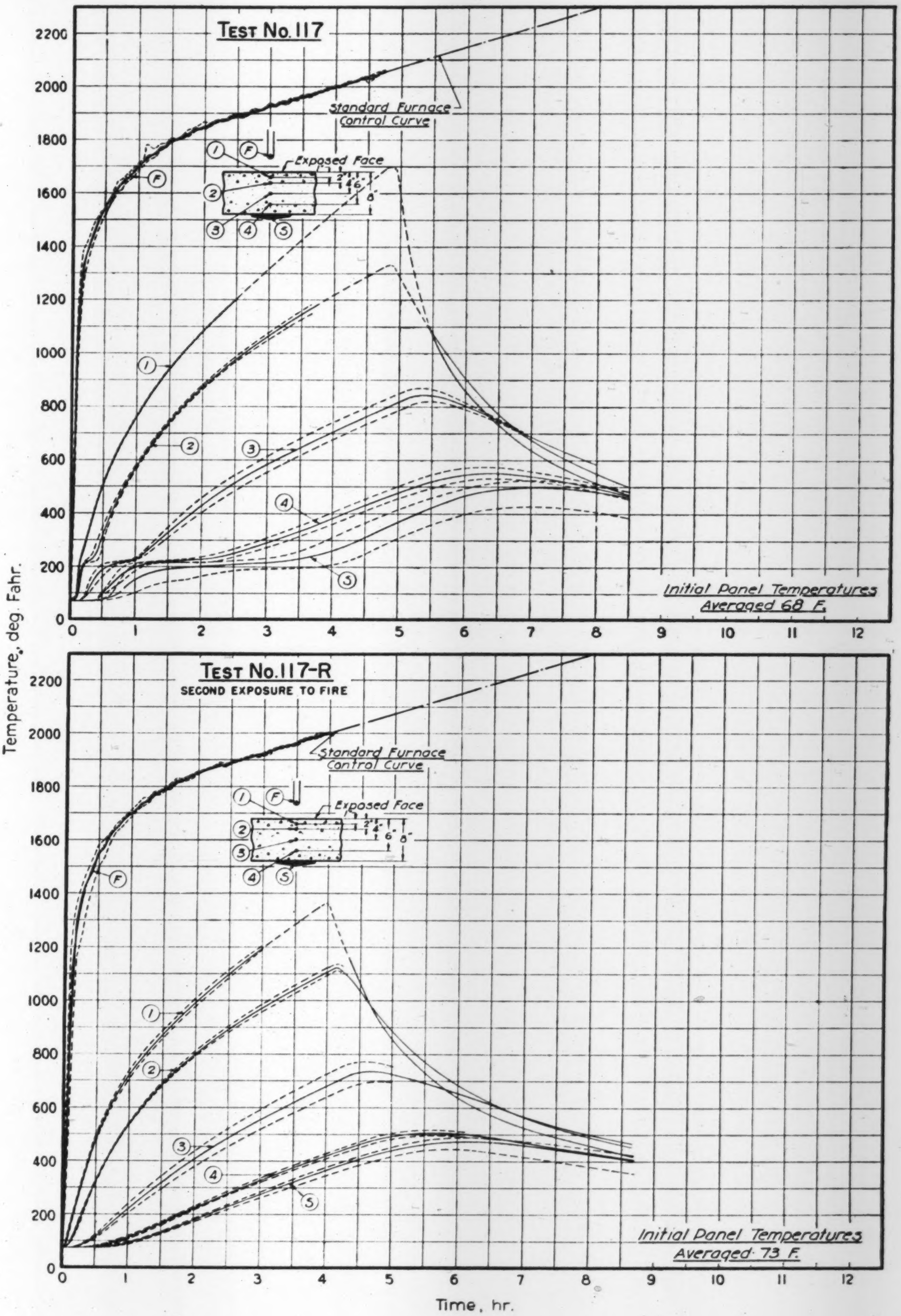

FIG. 18.—Time-Temperature Curves for Tests Nos. 117 and 117-R.

8-in. slab; siliceous gravel, graded 0 to ¾-in.; air-dried 13 months.

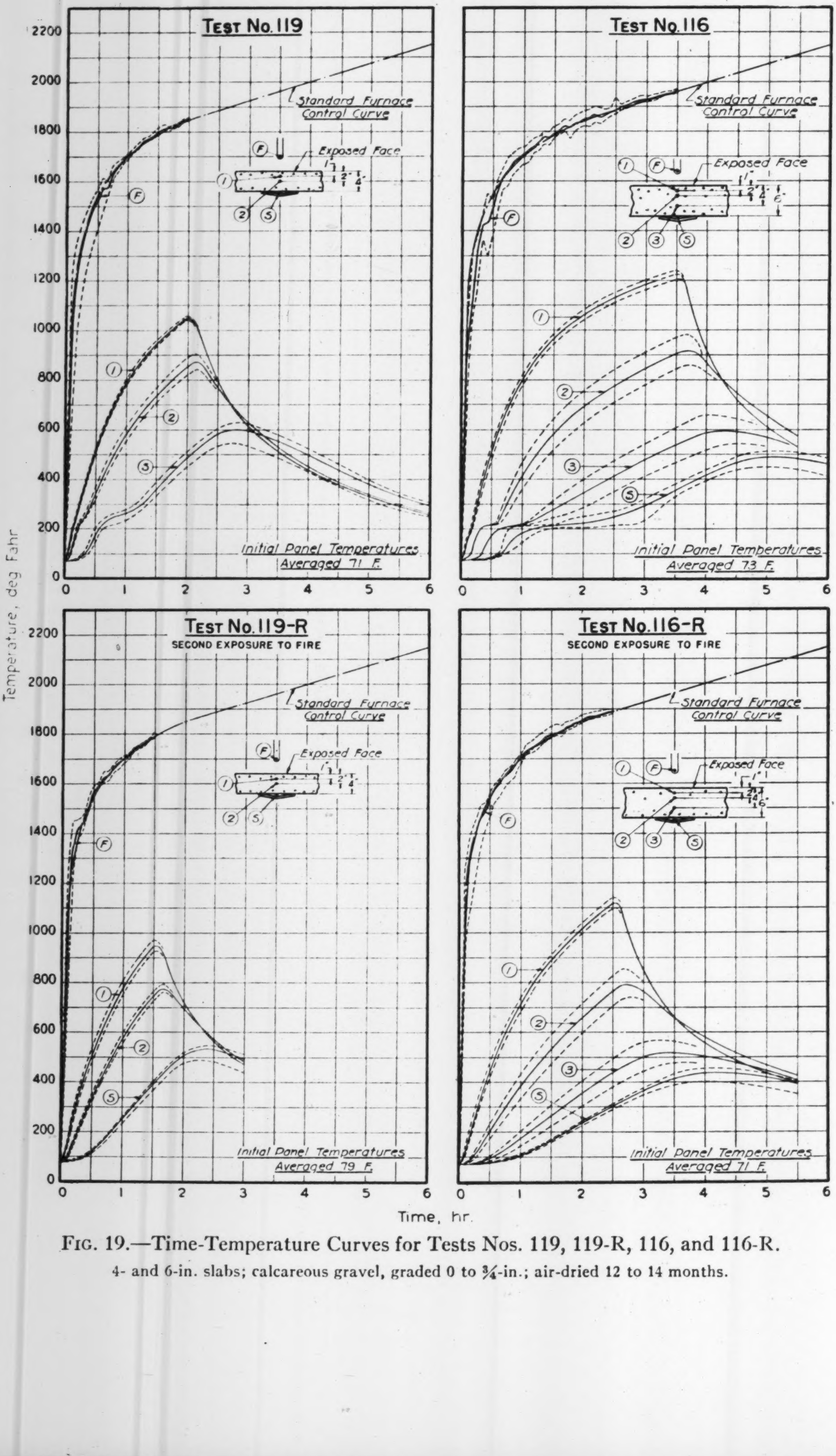

FIG. 19.—Time-Temperature Curves for Tests Nos. 119, 119-R, 116, and 116-R.

4- and 6-in. slabs; calcareous gravel, graded 0 to ¾-in.; air-dried 12 to 14 months.

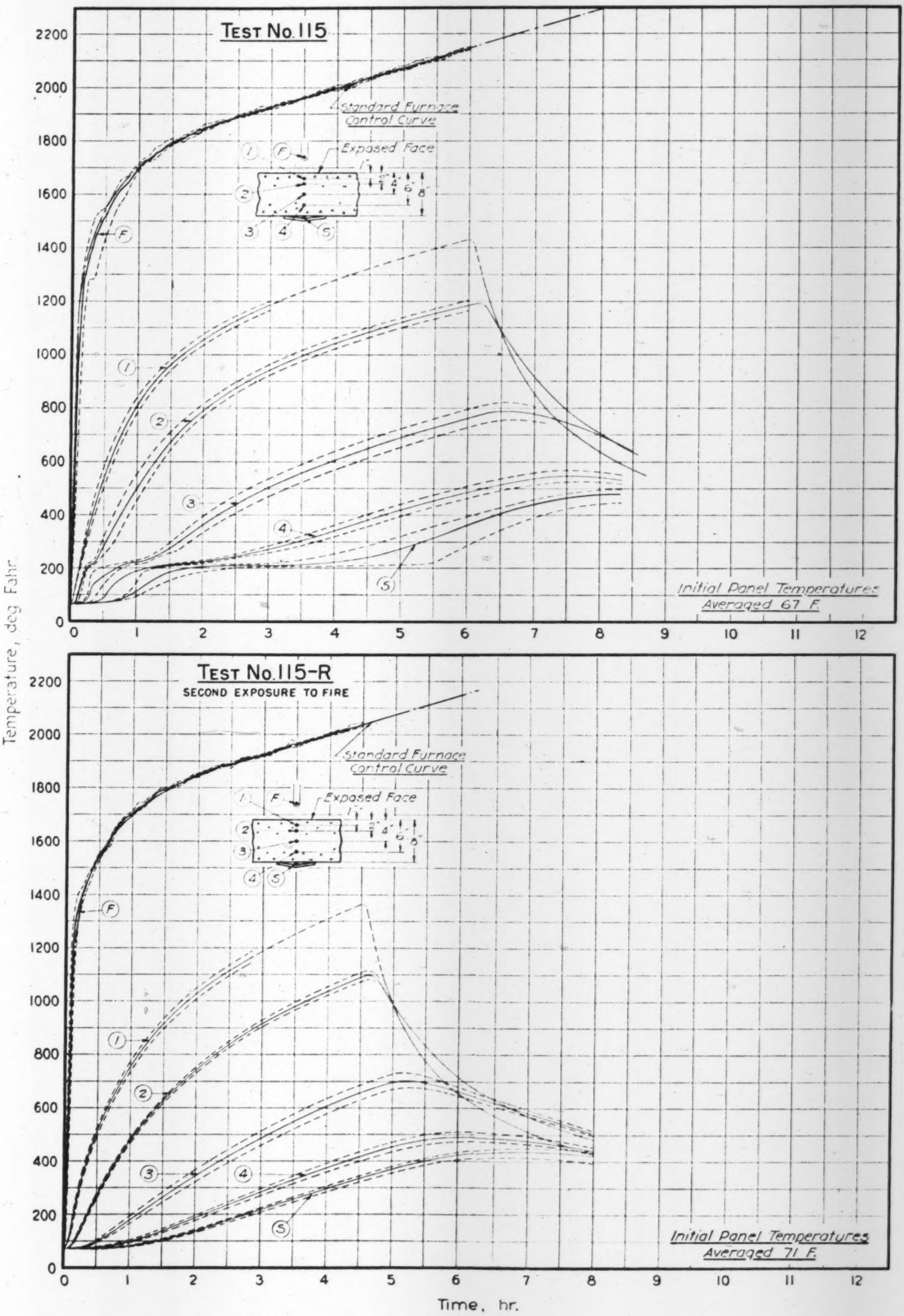

FIG. 20.—Time-Temperature Curves for Tests Nos. 115 and 115-R.

8-in. slab; calcareous gravel, graded 0 to ¾-in.; air-dried 13 months.

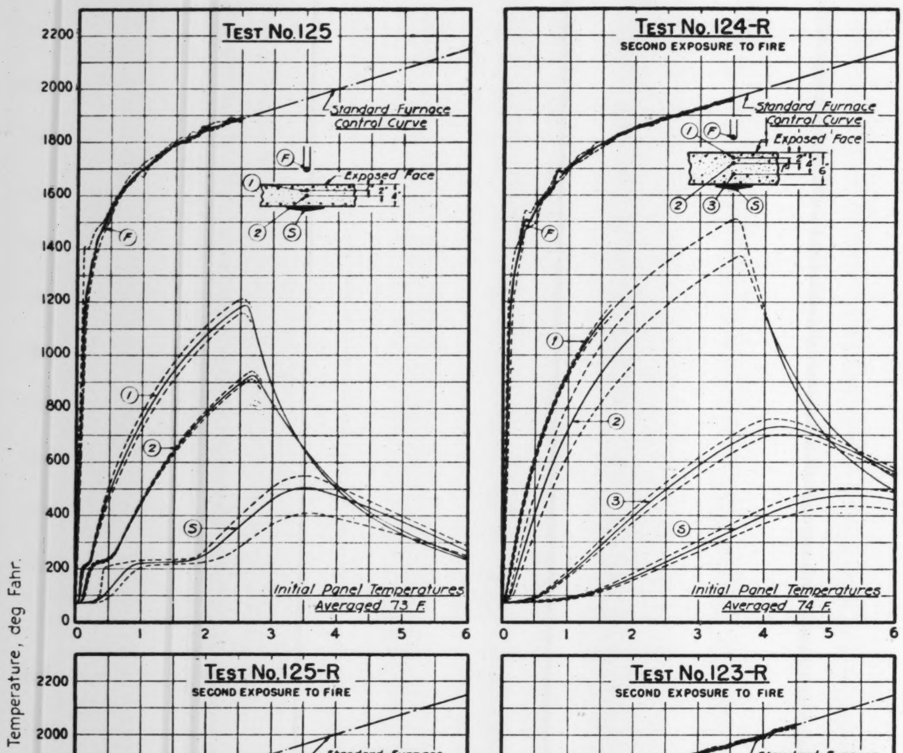

FIG. 21.—Time-Temperature Curves for Tests Nos. 125, 125-R, 124-R, and 123-R.

4-, 6-, and 8-in. slabs; Haydite, graded 0 to ¾-in.; air-dried 13 to 14 months.

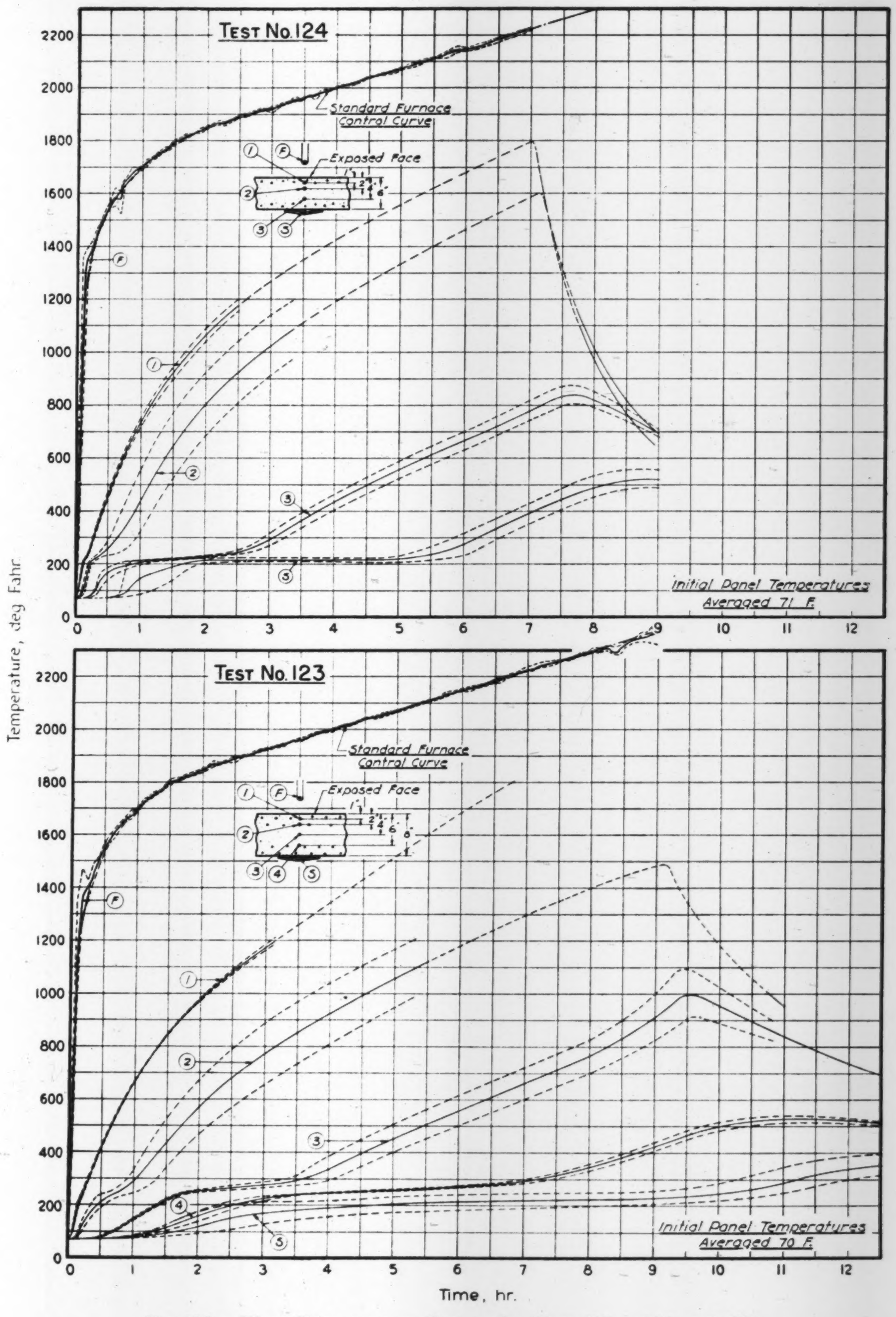

FIG. 22.—Time-Temperature Curves for Tests Nos. 124 and 123.

6- and 8-in. slabs; Haydite, graded 0 to ¾-in.; air-dried 13 to 14 months.

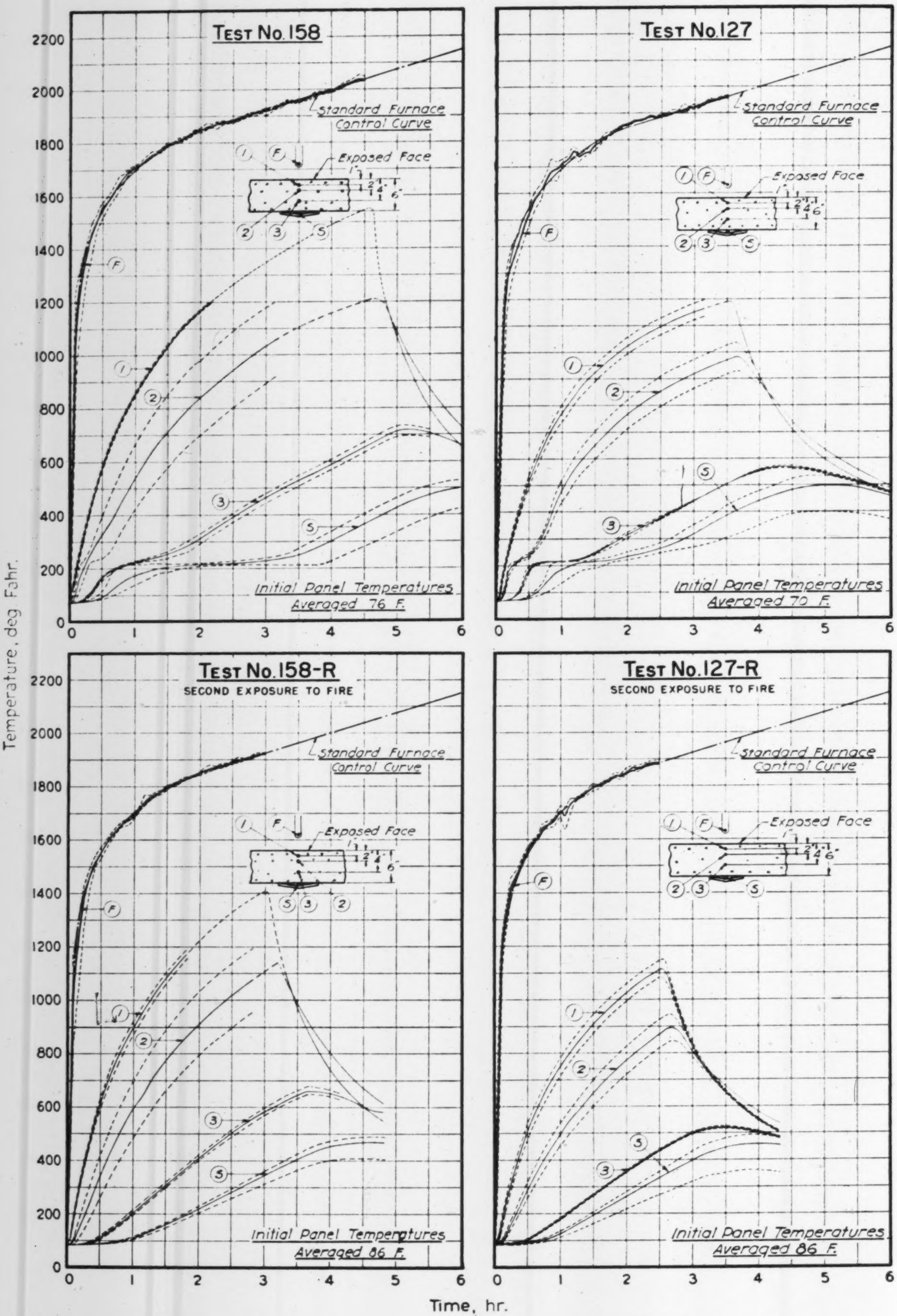

FIG. 23.—Time-Temperature Curves for Tests Nos. 158, 158-R, 127, and 127-R.

Tests Nos. 158 and 158-R.—6-in slab; mixture 0 to ¾-in. siliceous gravel with equal volume of 0 to ¾-in Haydite; air-dried 12 months.

Tests Nos. 127and 127-R.—6-in. slab; calcareous gravel, graded 0 to ¾-in.; air-dried 1 month.

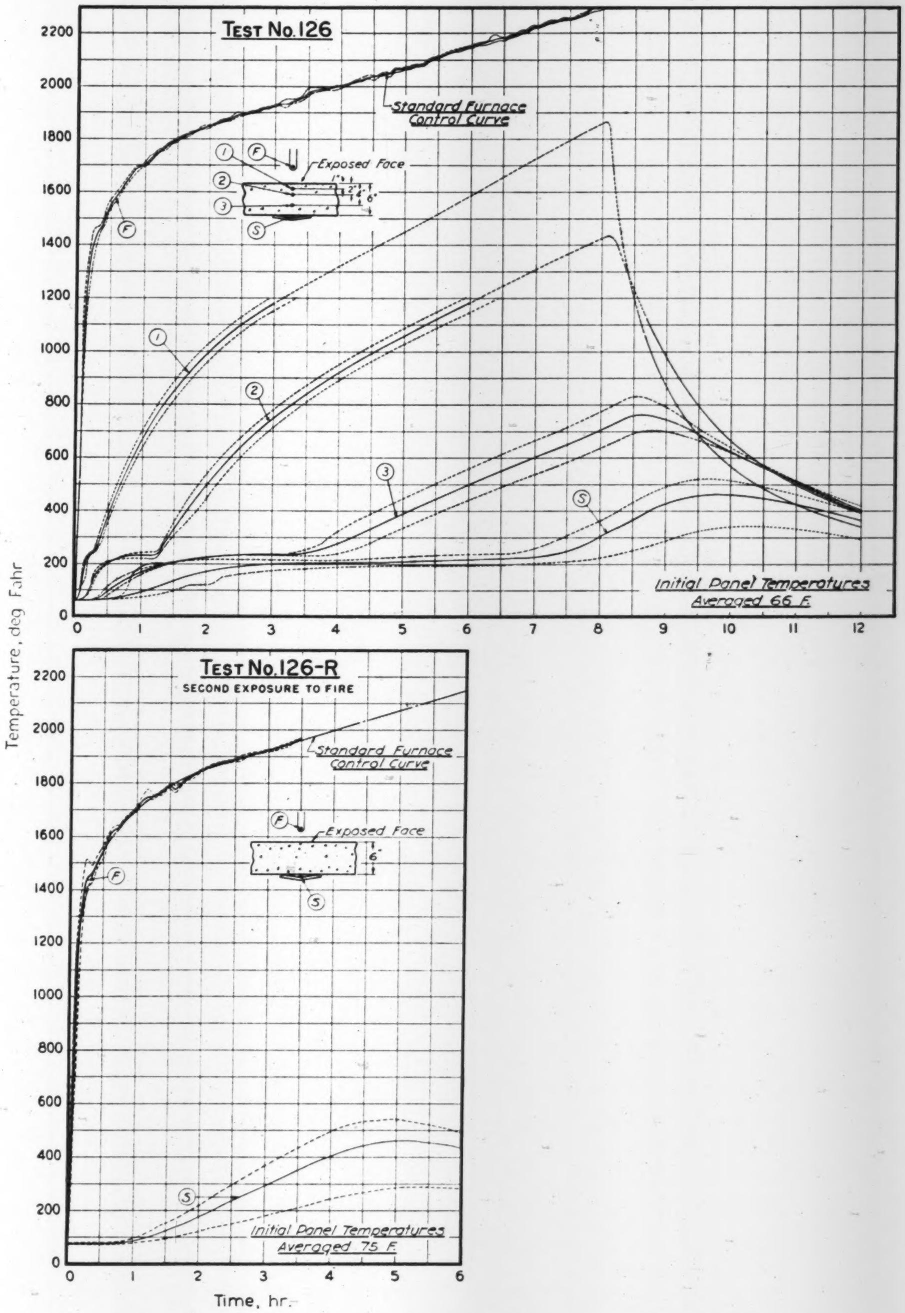

FIG. 24.—Time-Temperature Curves for Tests Nos. 126 and 126-R.

6-in. slab; Haydite, graded 0 to ¾-in.; air-dried 1 month.

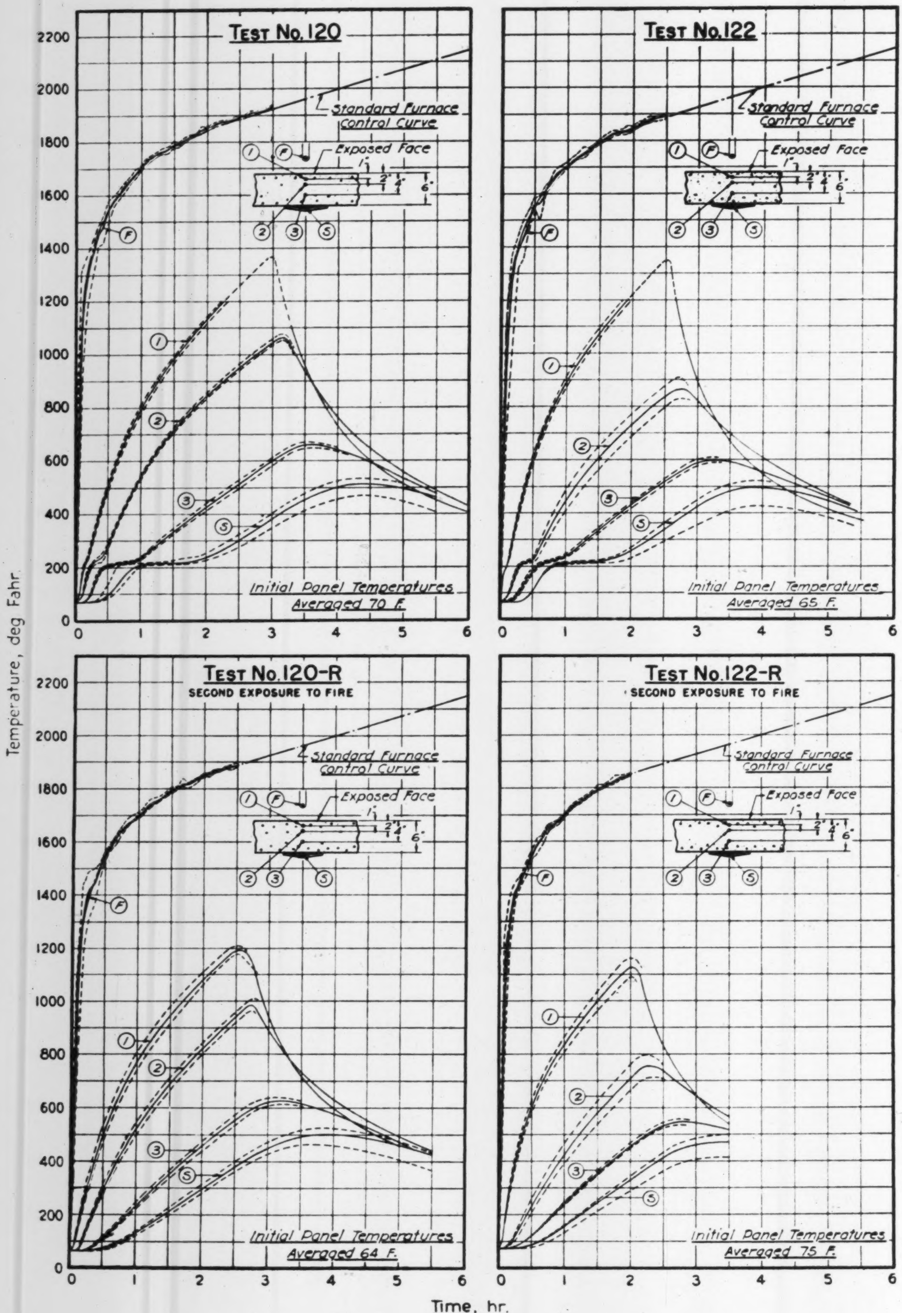

FIG. 25.—Time-Temperature Curves for Tests Nos. 120, 120-R, 122 and 122-R.

Tests Nos. 120 and 120-R.—6-in. slab; siliceous gravel, graded 0 to ⅜-in.; air dried 12 months.
Tests Nos. 122 and 122-R.—6-in. slab; siliceous gravel, graded 0 to 1½-in.; air-dried 12 months.

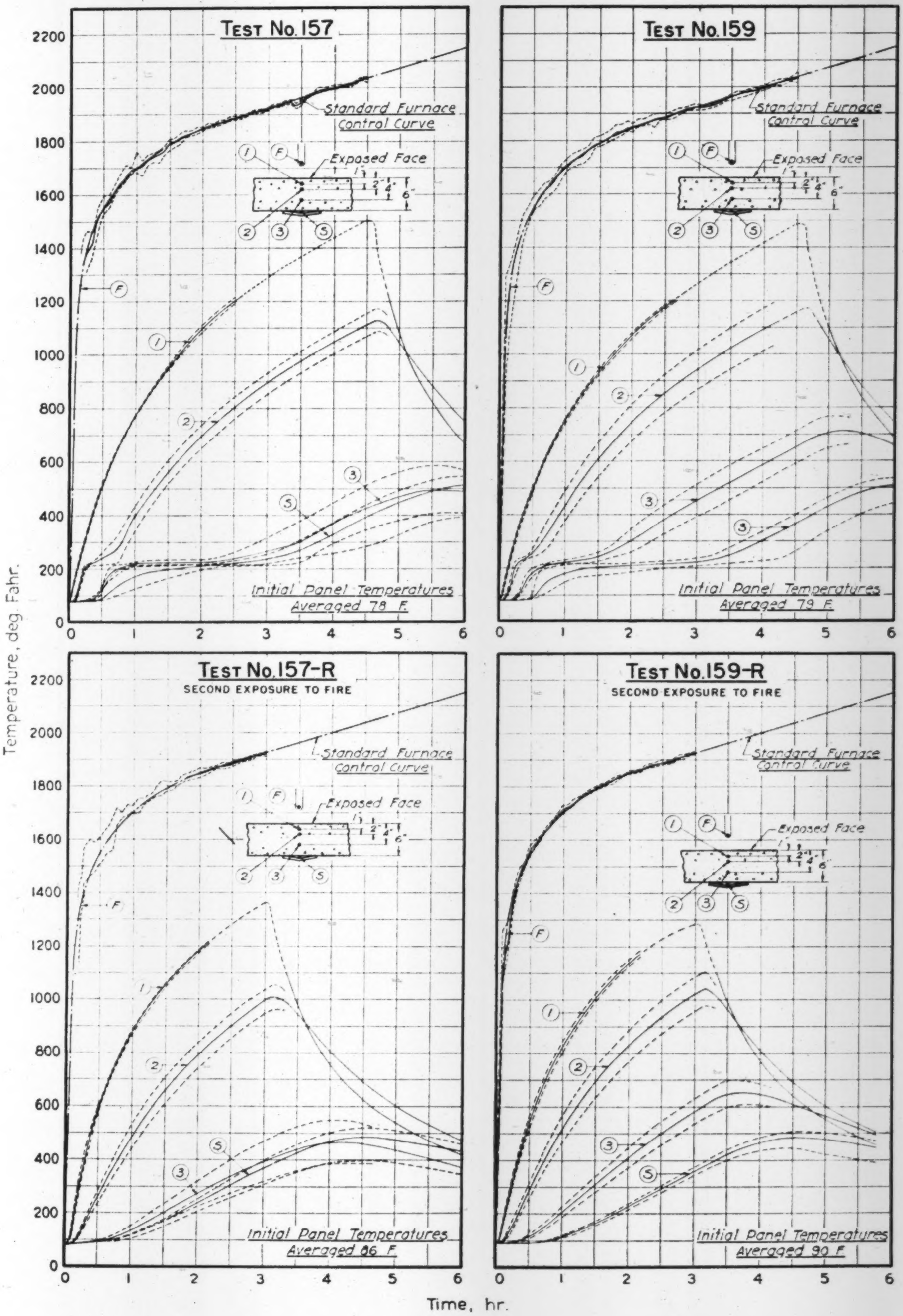

FIG. 26.—Time-Temperature Curves for Tests Nos. 157, 157-R, 159, and 159-R.

Tests Nos. 157 and 157-R.—6-in. slab; mixture 0 to ⅜-in. siliceous gravel with equal volume of 0 to ¾-in. Haydite; air-dried 12 months.

Tests Nos. 159 and 159-R.—6-in. slab; mixture 0 to 1½-in. siliceous gravel with equal volume of 0 to ¾-in. Haydite **air-dried 12 months.**

No. 18 gage couples the values would have been about 5 per cent higher than with the more sensitive No. 20 gage couples. Had the fire-endurance periods actually been based on the temperature rise indicated by the thermometers, the values would have averaged about 4 per cent higher. This can be seen from the time periods for the thermocouples and thermometers given in column 8 of Table I.

(*b*) In these tests the temperature-sensitive points of the furnace thermocouples were placed 3 in. from the exposed face of the slabs instead of 6 in. as is specified by the present standard. This practice was followed to maintain the precision of furnace control but had the general effect of subjecting the exposed face of the slabs to somewhat higher furnace temperatures (because the radiation loss from the thermocouple to the cool specimen increases as the distance between them is reduced) than would be obtained under present standard requirements, and therefore increased the rate of temperature rise on the unexposed face. Had the couples been placed 6 in. from the exposed face of the slab specimen it is believed that the fire-endurance period would have been about 3 per cent higher than with the 3-in. distance used. (For a discussion of these and other factors see Article 8A, page 68 of reference (**17**).

The more severe test condition described in (*c*) below would also tend to give values of the fire-endurance period below those obtained under the conditions which normally prevail in classification tests of large specimens:

(*c*) In order to maintain as uniform conditions as practicable in the furnace room from test to test, all doors and windows were kept closed during the conduct of each fire test, regardless of season. This minimized drafts and permitted more reliable observations relative to steaming, smoking, etc., but had the effect of hastening the temperature rise on the unexposed face and thus reducing the fire-endurance period of the test specimen. With the much greater side and headroom required for tests of standard specimens, the cooling effect of drafts is more pronounced particularly when entrances and exits to the furnace room are not closed.

Curves of Furnace and Slab Temperatures:

Figures 17 to 26 give the time-temperature curves for the first and second fire-endurance test of each solid slab specimen. All curves are based on temperature measurements with thermocouples as described in the notes and diagrams of Fig. 1. Average temperatures at each station are represented by full-line curves; maximum and minimum temperatures by dash-line curves.

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DISCUSSION

MR. S. H. INGBERG[1] *(presented in written form)*.—In commenting on this paper, I do not want to minimize the many valuable results and relationships that Mr. Menzel has obtained in these tests. My main comments have to do with the method of testing, and I shall discuss it almost exclusively from that standpoint.

In our fire test specifications we require certain minimum sizes of specimens in order to have a fairly standard test. These are comparatively large and represent an average, if not a maximum, of what will be used in the structure.

We adopted a provision a year or two ago whereby we can go to a lower size only if, under the conditions of application, that size will not be exceeded. However, the story heights in buildings are fixed by law and by practical considerations. I believe the lowest that is now permitted is 7½ ft. and the more usual story heights are 8 to 10 ft.

The present paper gives results of fire tests with wall specimens 5 ft. 4 in. wide and 6 ft. high tested under load. The details of the testing procedure are well observed and the exposure and other test conditions are well within the margin of deviation permitted by the test specifications. There are, however, limitations introduced by the specimen equivalent in thickness but considerably smaller in height and width than assemblies applied in buildings. If the required minimum sizes of test specimens were wholly arbitrary, the matter might be passed up as of minor importance, but results of fire tests do show decided differences for some materials because of size and border conditions. This refers mainly to the extent to which stability and integrity of structure are maintained as exposed to fire. In the introduction and conclusions of the paper, the possibility is indicated that these may limit the performance of constructions under fire exposure.

It appears that the results of the tests reported in this paper are subject to decided limitations as applied to full-size building assemblies where the concrete is made with siliceous aggregates and to less extent for concrete made with calcareous sand and gravel. This is due to the low height of the wall specimens tested with restraint only from the applied load which allowed almost full freedom for expansion at the borders. This gave conditions more favorable for stability of the walls and partitions than obtains in buildings even if of minimum permissible story height.

It can be shown that if an assembly, such as a wall supported at the top and bottom and free at the sides, is exposed to fire on one side, the center deflection, f, is given very nearly by the following formula:

$$f = \frac{CL^2(T_1 - T_2)}{8h}$$

where:

C = the coefficient of expansion of the wall material,

L = the height,

$T_1 - T_2$ = the temperature difference between the outer layer on the exposed and that on the unexposed side, and

h = the wall thickness.

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[1] Chief, Fire Resistance Section, National Bureau of Standards, Washington, D. C.

Accordingly, with other conditions the same, the deflection will vary with the square of the height and inversely with the thickness. The factor $C(T_1 - T_2)$ can be taken as representing the difference in extension between the outer fiber on the exposed and that on the unexposed side, since there are shrinkage as well as temperature effects, and the formula thus interpreted will hold in a general way for the combined effect of the two. For constructions restrained at the borders, there is a further deflection from the extension of the axis of the wall.

The stability of a wall under load or restraint depends on the eccentricity of the thrust at the borders with reference to the section of the wall having the greatest deflection. While in the absence of tensional resistance in the materials and resisting moments at the borders, the limit of stability is reached when the line of thrust comes outside of the middle third of the wall thickness, actually these other effects, including also shift of the line of thrust toward the fire-exposed side as indicated in Fig. 5 of the paper, permit a higher deflection before collapse occurs. The deflections of walls as exposed to fire are high as compared with one sixth of the wall thickness, and hence failure on this score can occur for walls made of materials exhibiting relatively high expansion under heat.

In comparison with the author's results for 4, 6, and 8-in. walls of monolithic concrete made with siliceous sand and pebble aggregates, we obtained collapse because of excessive deflection for a 4-in. wall 10 ft. high tested under load of 100 psi., before the end of the first test hour. The concrete was ready-mixed of approximately 1:2.6:3.2 proportion by volume and of 6 to 7 in. slump. Reinforcement of 4 by 4 in. mesh of No. 6-gage steel wires was provided in the middle of the wall. The deflection at 35 min., before it was appreciably influenced by the impending failure, was near $3\frac{1}{2}$ in. This deflection is obtained from the above formula for coefficient of expansion 0.0000067 and temperature difference of 1170 F.

In another test with a similar wall, 10 by 16 ft., restrained on all edges, no collapse occurred before the limit of temperature rise obtained on the unexposed side, when the deflection was 9 in. With the vertical edges free, it would apparently have collapsed before this time.

In a test with a similar 6-in. thick wall under load of 300 psi., the limiting temperature rise on the unexposed side was reached when the maximum center deflection of the wall was about 4 in. The test was continued until a maximum center deflection of 5 in. was obtained without failure due to load or deflection. As computed by the above formula and constant, the 5-in. deflection corresponds to a higher temperature difference between the two sides than could have obtained, indicating possible effects from extension of the axis of the wall under partial restraint from the applied load. This also obtained in a test of a similar 8-in. thick wall if the deflection at the mid-point of the wall is considered. If taken as an average for the mid-height at the center and quarter points in the width of the wall, the deflection would correspond to the computed figure within the limit to which temperature difference and coefficient are known. These tests did not indicate any noticeable effect from shrinkage of the fire-exposed concrete, the increase in deflection being roughly linear with time after the first one half or one hour.

We have so far not conducted fire tests of solid walls of concrete made with other than siliceous aggregates. We have, however, made tests of walls built of prefabricated concrete units made with a range in aggregates. As made with siliceous sand and gravel, collapse within the first half-hour of the test was obtained with a 4-in. partition tested restrained and within the first hour as tested plastered with $\frac{1}{2}$ in. thickness of gypsum plaster. The same applies for 8-in. walls having a single 3-core unit in the wall thickness and tested under load of 80 psi. of gross area. A similar wall of 12-in. thickness failed from lack of stability within the first half hour but as plastered or if made with two units of 4 and 8-in. thickness, respectively, there was no collapse and the fire resistance end point was determined by temperature rise on the unexposed side after 5 hr. A 4-in. partition of units made with calcareous sand and gravel and tested restrained collapsed within the first half hour but satisfactory stability was indicated for a similar partition plastered on both sides. Satisfactory stability was also indicated for unplastered partitions and walls of 4 in. or greater thickness built of units made with cinder, slag, expanded burned clay, and pumice aggregates. These constructions were all tested on a height of 10 ft.

The term "siliceous aggregates" should be taken to include those made up largely of pebbles and grains of quartz, chert, flint or other uncombined forms of silica. A chemical analysis may give 60 per cent or more of silica for aggregates in which feldspar, granite, and volcanic minerals such as rhyolite and andesite predominate, the presence of which would be indicated by the associated content of sodium, iron, and aluminum oxides. The performance under fire exposure with these minerals is in general more favorable than with the given forms of uncombined silica. With this understanding, the above results are in no apparent conflict with those obtained by other laboratories in fire tests of concrete masonry walls of units made with natural aggregates as well as with the cinder, slag, and burned-clay materials.

In the tests of the solid concrete walls, as of those built of prefabricated units, there was no spalling of concrete made with siliceous aggregates, and this can be attributed to lack of full restraint. Further information on the effect of the latter condition was obtained in fire tests of floor slabs. With unrestrained but loaded reinforced slabs tested in the pilot furnace with exposed area of 4 by 8 ft., no spalling of concrete was obtained for slab thickness from 4 to 8 in. In the large furnace, accommodating specimens $13\frac{1}{2}$ by 18 ft., there was also no spalling with a 6-in. slab supported on beams having a cantilevered free end beyond a girder support, but failure on this score was obtained with a 4-in. and a 6-in. reinforced slab more fully restrained. These constructions were well seasoned before testing. The aggregates for the concrete of these slabs as for the solid concrete walls were Potomac River sand and gravel containing 90 per cent or more of quartz, chert, and flint.

Monolithic reinforced concrete floor slabs of any considerable size induce restraint on areas thereof subjected to fire exposure, that may result in disruptive effects limiting the fire resistance of the construction below what would otherwise be obtained. We do not have sufficient data to indicate the limit of area below which these disruptive effects from expansion would not be expected. This may be such that for a floor in a dwelling, some 20 by 30 ft., the expansion effects might be minimized. We have noted in fires in large commercial buildings of reinforced concrete made with high-silica aggregates that the spalling is much less decided in the outer than in interior panels, apparently because of less restraint. All that can be stated with some certainty is that where the slab is of some considerable size the restraint imposed by its monolithic structure and its reinforcement will induce such effects, whereas it was not obtained with unrestrained or partially restrained slabs of the size accommodated in our test furnaces.

Another detail of the testing procedure to which the author makes reference is the size of wire for thermocouples indicating temperatures on the unexposed side under the 6 by 6-in. asbestos pads. With the maximum No. 18 gage (0.04 in.) size permitted, the immersion under the pad would be about 70 diameters. Computations on the possible error involved from conduction to the couple by the wire indicate it to be 1 F. or less. The theoretical relations are complex and certain assumptions must be made but within a reasonable range in assumptions this appears to be the order of possible error. This is in general within the range of uniformity or calibration of the thermocouple wire or in fact of the other instruments and conditions involved in the measurement. Recent comparative temperature determinations made with No. 18 and No. 22 gage straight and No. 18 gage coiled thermocouple wire, lead to similar conclusions. In our tests we use No. 22 gage wires separated and coiled under the pads.

MR. CARL A. MENZEL[2] (*author's closure by letter*).—The author wishes to express his appreciation to Mr. Ingberg for his remarks and suggestions based on the results of fire tests on large concrete walls at the National Bureau of Standards.

The tests cited by Mr. Ingberg were made on walls 16 ft. wide and 10 ft. high whereas it has been the procedure of Underwriters' Laboratories to test walls 10 ft. wide and 11 ft. high, and the author's procedure to test walls $5\frac{1}{3}$ ft. wide and 6 ft. high. Although the author's walls were smaller than the 100 sq. ft. total area and 9 ft. minimum dimension specified in A.S.T.M. Standard Methods of Fire Tests of Building Construction and Materials (C 19 – 41) for his tests of load-bearing walls, Mr. Ingberg's walls were 6 ft. wider or 60 per cent larger in width and total area than required by these specifications. The walls tested at Underwriters' Laboratories conform most closely to the A.S.T.M. standard although they were 1 ft. higher (10 per cent larger in height and total area) than required.

Although the results of the author's tests on walls $5\frac{1}{3}$ ft. wide by 6 ft. high may be somewhat more favorable as regards load-carrying capacity and stability than would be expected from walls of full story height (a point which the author has taken into account in various sections of his paper) he believes that the results of Mr. Ingberg's tests on walls 16 ft. wide are open to some question because they probably introduced load concentrations and deflections greater than intended in standard size specimens, and greater than can be expected in bearing walls under actual service conditions.

With the method of loading employed by Mr. Ingberg, by Underwriters' Laboratories, and by the author, there would be a greater load concentration at the corners [illustrated in Fig. 5 (*c*)] for walls 16 ft. wide than for either standard walls 10 ft. wide tested by Underwriters' Laboratories, or the walls $5\frac{1}{3}$ ft. wide tested by the author. Moreover, this load concentration, which results from the change in shape of the wall during fire exposure, is believed to be more pronounced within the rigid test frame of the fire test equipment than would occur when the same wall is exposed to fire while carrying a uniformly distributed bearing load under actual service conditions. For example, if the wall in service is carrying the uniformly distributed load imposed by a floor slab this slab will deflect or bow toward the fire and thereby tend to maintain contact with the curved bearing edge at the top of the wall instead of developing the separation between wall and test frame shown in Fig. 5 (*c*). As a result of this bowing, the development of load concentrations at the upper corners is automatically minimized and walls may actually be less subject to collapse under service conditions than when they are loaded in the manner followed by Mr. Ingberg, Underwriters' Laboratories, and the author.

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[2] Associate Engineer, Research Laboratory, Portland Cement Assn., Chicago, Ill.

The degree of load concentration, or departure from uniformly distributed loading, obtained in fire tests is not subject to close analysis but it appears that it will increase with the width of the wall, the rigidity of the test frame and the rigidity of the loading beam. Moreover, if the thrust of the hydraulic jacks is transmitted to the wall by means of two loading beams (photographs of wall specimens show that Mr. Ingberg employs two short beams to load this 16 ft. wide wall), the degree of load concentration would appear to be greater than if one loading beam is used. This is because there will be a lesser tendency for two half-length beams to conform to the curved bearing edge of a wide wall than a single full-length beam of the same cross-section.

The preceding remarks are offered to show that the test conditions vary in different laboratories and may inadvertently introduce factors and differences which are not present in full-size bearing walls in service. They appear to merit some consideration because they may determine whether a wall is able to adjust itself and remain stable at a critical stage of the fire test.

In tests of 6- and 8-in. slabs of solid concrete with highly siliceous aggregate, Mr. Ingberg obtained deflections greater than could be accounted for by temperature differentials or by loads and temperature differentials combined. The large deflections were evidently caused by artificial restraints and load concentrations, yet it appears that these walls did not fail to carry a superimposed load of 300 psi.

The author considers that bearing walls are usually unrestrained at the end edges and therefore tested his walls without imposing end restraint. It is interesting to note that in a test of a 4-in. solid slab (10 ft. high and 16 ft. wide) of highly siliceous aggregate, Mr. Ingberg ascribes lack of collapse with a large deflection (9 in.) to the support resulting from restraint at the vertical edges. Without this restraint a similar wall collapsed under a load of 100 psi. before the first hour. These results suggest that it is possible for restraint to be helpful whereas in other tests cited by Mr. Ingberg he indicated that restraint was harmful.

The amount of artificial restraint that should be imposed in tests of non bearing walls (simulating walls built between columns and floor beams) or of loaded floor slabs (simulating the interior panels of a floor area) is difficult to determine and is a matter which deserves the consideration of A.S.T.M. Committee C-5 on Fire Tests of Materials and Construction so that the test procedure covering this point will be adequate but not unreasonably severe.

As pointed out in the paper, the author was well aware of the limitations of tests on wall specimens smaller than the standard requires. However, he feels that the results obtained in a systematic research on a total of 231 of these smaller walls have provided reliable information on the effect of a wide range of variables. Thus far there have been no instances in which the indications of the tests on small walls of hollow concrete masonry units have not been confirmed by tests at Underwriters' Laboratories on large 10 by 11-ft. walls of hollow units. From this it might be inferred that the tests of smaller solid walls reported by the author would perform at least as well in standard size walls as did the walls of hollow masonry units. In the many tests made on hollow concrete masonry walls at the Underwriters' Laboratories since 1921, not a single wall has failed to carry its normal working load of 80 psi. of gross bearing area or has collapsed when restrained at all four edges. These Underwriters' Laboratories' tests were made on 8- and 12-in. thick walls with a single hollow unit through the wall thickness. The units were made from natural aggregates such as calcareous and siliceous sand and gravel, crushed limestone, granite, traprock and from artificial aggregates such as slag, cinders, and Haydite. Some of the earlier tests were made on composite walls restrained on all edges but the effect of the highly siliceous aggregate used in units comprising the upper quarter of the wall was modified by the effect of cinder, slag, and limestone used in units comprising the three lower quarters. No tests were made later on walls consisting entirely of highly siliceous aggregate because the trend of development was in the direction of units made with the light-weight artificial aggregates. It should be noted that in Mr. Ingberg's tests on large walls 4 in. or greater in thickness built of units made with cinder, expanded slag, expanded burned clay, and pumice aggregates, all the walls have shown satisfactory stability. Furthermore, that all walls built of units made with sand and gravel aggregate (both calcareous and highly siliceous) showed satisfactory stability when protected with plaster.

The author is unable to comment further on the tests conducted by Mr. Ingberg since complete data concerning them have not yet been published.

ACCELERATED WEATHERING OF BITUMINOUS MATERIALS—EFFECT OF OPERATING VARIABLES

By Bruce Weetman[1]

Synopsis

Accelerated weathering tests were made on five roofing asphalts from different crude oil sources in which the operating variables of the test were varied in order to determine their effect on the test results.

The data obtained show that the test temperature is of prime importance and must be carefully selected and rigidly controlled in order to obtain reproducible results. Amperage, refrigeration temperature, force of the washing water, and surface cleanliness of the aluminum panel do not effect the test results. The use of the electrometric procedure for determining panel failure is necessary for those asphalts which fail by the opening of fine cracks or "pin points."

It is recommended that a temperature of 140 F. be used in this test in order adequately to differentiate between the weathering qualities of different asphalts in the shortest practical time.

The service life of prepared roofing, as well as built-up roofs, depends to a large extent upon the weathering characteristics of the asphalt which goes into their construction. A satisfactory roofing asphalt must be able to withstand the deteriorating effects of the elements over an extended period of time. Since the roofing manufacturer guarantees his roofs for a period of several years it is essential that he know in advance the weathering characteristics of the asphalt that he is using as it is obvious that he cannot afford to wait a period of years for service data in order to control the quality of his manufacture. Neither can the asphalt producer wait for field data to determine the weathering characteristics of his asphalt, as by that time the original crude-oil stocks may no longer be available. The necessity of having a dependable accelerated test for measuring the weathering characteristics of asphalts is apparent.

In spite of the fact that accelerated weathering tests had been made on asphalts for a number of years, the first published method was by Strieter in 1933 (**2**).[2] He took the first steps in standardizing a procedure for determining the accelerated weathering qualities of asphalts employing the use of an Atlas Weatherometer which was available at that time in most roofing and asphalt laboratories. Strieter's recommended procedure was adopted by most laboratories who realized the desirability of having a standard method instead of the numerous individual ones previously used. The operating variables in the original procedure were not controlled rigidly enough to guarantee different laboratories obtaining comparable results, and consequently the test was relatively unsatisfactory. However, it did possess value as a laboratory tool for the separation of good and poor weathering asphalts. Accelerated weathering results obtained by four laboratories using this procedure on a typical coating asphalt are as follows:

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[1] The Texas Company, Beacon, N. Y.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1161.

Laboratory	Accelerated Weathering Cycles to Failure	Variation from Minimum, per cent
A.........	25	0
B.........	28	12
C.........	32	28
D.........	43	72

The variations as high as 72 per cent were due to the operating variables in the test, as all the panels were made at one laboratory at the same time and from the same sample of asphalt. This procedure has been modified as described in A.S.T.M. Tentative Recommended Practice for Accelerated Weathering Test of Bituminous Materials (D 529 – 39 T).[3]

No further standardization of the test can be made until the effects of several operating variables have been determined. With this in mind, a series of tests were initiated which were designed to show the effect of temperature, amperage, refrigeration temperature, methods of examining the panels for failure, and other factors upon the accelerated weathering of bituminous materials.

APPARATUS AND METHOD

The accelerated weathering unit used in this work was essentially the same as the Atlas Weatherometer. The difference was in the drum and tank assembly. The drum was made of extra-heavy copper screening instead of aluminum sheet and revolved at a rate of 1 rpm. This drum was located in a 40-in. diameter copper tank. The panels were supported on the screen by means of clips instead of by panel holders.

The following cycle of weathering was used throughout the experimental work:

8.00 a.m. to 8.45 a.m...............0.75 hr.—Inspection
8.45 a.m. to 10.30 a.m..............1.75 hr.—Refrigeration
10.30 a.m. to 10.45 a.m.............0.25 hr.—Interval
10.45 a m. to 11.45 a.m.............1.00 hr.—Water spray
11.45 a.m. to 12.00 a.m.............0.25 hr.—Interval
12.00 a.m. to 1.30 p.m..............1.50 hr.—Light
1.30 p.m. to 1.45 p.m...............0.25 hr.—Interval
1.45 p.m. to 3.45 p.m...............2.00 hr.—Water spray
3.45 p.m. to 4.00 p.m...............0.25 hr.—Interval
4.00 p.m. to 8.00 a.m..............16.0 hr.—Light

Failure was defined as the number of cycles elaspsed before the aluminum of the base panel became visible in two locations more than ¼ in. from the edge of the panel.

TABLE I.—PHYSICAL PROPERTIES OF ASPHALTS USED.

	Asphalt No. 1	Asphalt No. 2	Asphalt No. 3	Asphalt No. 4	Asphalt No. 5
Specific gravity at 77 F......	0.994	1.032	1.024	1.035	1.041
Softening point, ring-and-ball, deg. Fahr............	212	219	224	226	220
Penetration at 77 F.........	22	14	17	16	16
Penetration at 32 F.........	14	9	11	11	11
Penetration at 115 F........	33	25	31	30	29

A standard procedure for making the asphalt panels was established at the beginning of this work so as to eliminate any discrepancies which might result from this phase of the test. In this procedure, the temperature of the asphalt, trimmer knife, and panel holder were held at predetermined temperatures of 325 F., 425 F., and 200 F., respectively, with an accuracy of ±10 F. The time for each step in the procedure was also controlled.

ASPHALT STOCKS

In order to obtain as comprehensive a picture as possible as to the effect of variables in the test, five asphalts were selected for this work. Tests on these materials are shown in Table I. Each of these asphalts was made from a different asphaltic crude oil and with the exception of Asphalt No. 1 all have been accepted as satisfactory coating asphalts by the roofing industry. All asphalts were tested in duplicate under each test condition in order to minimize discrepancies.

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[3] 1942 Book of A.S.T.M. Standards, Part II, p. 1335.

TESTING VARIABLES

Temperature:

From our general knowledge of the low-temperature oxidation characteristics of petroleum hydrocarbons, it appeared that the operating temperature should play a marked rôle in the accelerated weathering test. Three runs were made on the five asphalts mentioned above at temperatures of 130 F., 140 F., and 160 F. These temperatures were measured with an A.S.T.M. thermometer E 1 (20 F - 39)[4] the bulb of which had been coated with asphalt and which was at panel distance from the arc. The temperature was controlled by a bimetallic thermostat located just inside the rows of panels. This thermostat was connected to a relay switch which started a fan blowing air into the weatherometer whenever the temperature exceeded that desired. The recorded temperature was adjusted to coincide with that shown by the thermometer.

TABLE II.—EFFECT OF TEMPERATURE ON ACCELERATED WEATHERING RESULTS.

	Cycles to Failure		
	130 F.	140 F.	160 F.
Asphalt No. 1	115	67	42
Asphalt No. 2	59	39	21
Asphalt No. 3	50	39	22
Asphalt No. 4	47	44	22
Asphalt No. 5	45	51	25
Average	63	48	26

The accelerated weathering results obtained for each asphalt at the above temperatures are shown in Table II.

These data show very definitely the dependency of the accelerated weathering test results on the operating temperature, a plot of the average results for the five asphalts at each temperature showing practically a straight-line decrease with increase in temperature. Individual asphalts were affected to different degrees by the 30 F. change in temperature, and this was sufficiently great to reverse the order of weathering for some of the asphalts investigated. Increasing the temperature to 160 F. not only reduces the number of cycles required to obtain failure to a very low value but also decreases the significance of the results by showing great similarity for most samples.

It was originally planned to obtain data at 170 F., but it was found that some of the asphalts showed flowing tendencies at that temperature and the run could not be made.

The data obtained in this investigation show that the 180 F. radiation temperature in the A.S.T.M. Tentative Recommended Practice D 529 is too high, both from the standpoint of causing some asphalts to flow on the panel, and of producing results which would be so similar as to prohibit differentiating between the weathering characteristics of products made from different crude oil sources or by different methods. Accelerated weathering differences between asphalts obtained at 180 F. may also be the reverse of actual field data due to the temperature being considerably above the average daytime roofing temperature in service.

In the absence of correlative data on accelerated and service weathering tests, it is recommended that the accelerated weathering test be made at an average temperature of 140 F. This is considered the maximum temperature which will differentiate the weathering characteristics of different asphalts without prolonging the testing period unnecessarily. This temperature should be measured by placing an A.S.T.M. thermometer E 1 (20 F - 39),[4] the bulb of which is coated with a $\frac{1}{16}$ in. film of asphalt, $\frac{1}{4}$ in. in front of the panels and at a point midway between the two rows of panels. This average temperature should be thermostatically controlled within ±2 F. by means of a suitable medium, and any variation from this value should be compensated for as the testing progresses.

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[4] Standard Specifications for A.S.T.M. Thermometers (E 1 - 42), 1942 Book of A.S.T.M. Standards, Part II, p. 974, also Part III, p 818.

Amperage:

The accelerated weathering of asphalts is essentially a low temperature photochemical oxidation reaction promoted by ultraviolet light (3). It would seem logical that as more electrical energy is put into the arc that a more intense light would be emitted which would in turn result in faster weathering of the asphalt. In order to investigate this particular variable, runs were made at a constant temperature of 140 F. but with currents of 15, 16, and 17 amp. It was considered desirable to express this variable as amperage in preference to power consumption since the previously mentioned tentative procedure refers to the arc amperage.

The amperage in the Atlas Weatherometer system is by no means constant but is characterized by short fluctuations. It varies over a wide range depending on the gap between the two carbons. Since our unit was not equipped with a recording ammeter it was necessary to devise some other means for determining the average amperes over any given period of time. This was accomplished by placing a sensitive watt-hour meter in the circuit so as to measure only the power consumption of the arc. By observing a large number of simultaneous voltage and current readings over a sufficiently long period of time for which the power consumption could be accurately measured, it was possible to calculate the power factor for the carbon arc light. Knowing the power factor and the line voltage it was then possible to predetermine what power consumption was necessary to obtain any desired amperage. The accelerated weathering unit was then operated so as to produce a predetermined power consumption which guaranteed the amperage desired. If the power consumption ran high during one cycle of testing, it was conpensated for by running the following cycle correspondingly low. By this means of operation it was possible to complete a run and maintain a desired average amperage very accurately.

The effect of amperage upon the ultraviolet light content of the emitted light was determined by a method developed by Anderson and Robinson (1). This method is based upon the fact that oxalic acid, when sensitized with uranyl sulfate, decomposes upon exposure proportionally to the amount of ultraviolet light present. The procedure consists of exposing 25 ml. of standard oxalic acid-uranyl sulfate solution to the arc light for 16 hr. and then back-titrating with a standard potassium permanganate solution to determine the amount decomposed. The exposure cell was made from a 1-in. diameter glass tube which was made light tight with black paint except for one end which was covered with a metal disk having a 0.312-in. diameter hole in the center. The cell was mounted in the weatherometer so that the exposure end was at panel distance from the arc. This procedure gives relative amounts of ultraviolet and not the actual percentage present in any spectrum.

The effect of increasing amperage upon the accelerated weathering results and the amount of oxalic acid decomposition is shown graphically in Fig. 1. In spite of the fact that the increasing amperage did not effect the accelerated weathering results, a decided increase in oxalic acid decomposition was obtained.

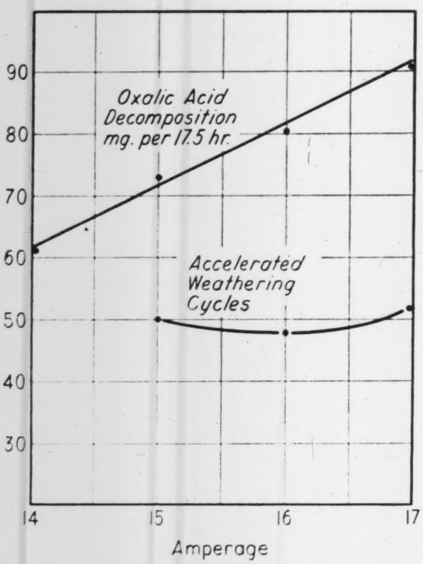

FIG. 1.—Effect of Amperage on Accelerated Weathering and Oxalic Acid Decomposition.

It can be concluded that the ultraviolet light acts as a catalyst in this weathering reaction and that it is not necessary to control the average amperage within the range of 15 to 17 with any great accuracy during the test. It is recommended, however, that the test be made at normal amperages in view of the narrow range investigated.

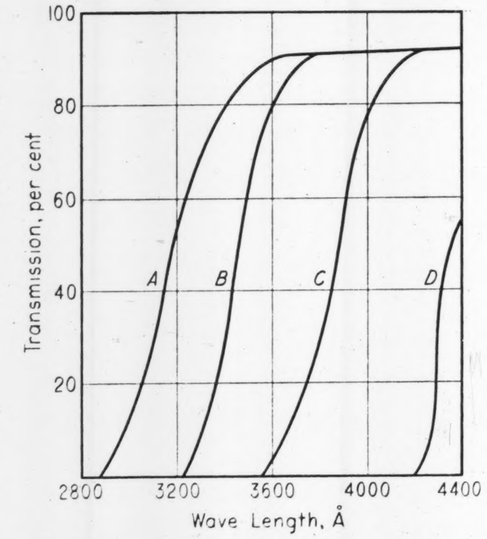

FIG. 2.—Transmission Characteristics of Filters.

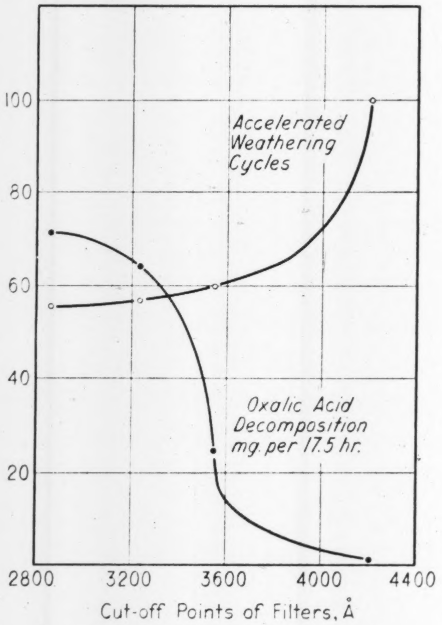

FIG. 3.—Effect of Light Filters on Accelerated Weathering and Oxalic Acid Decomposition.

Wave Length of Light:

In order to investigate the effect of the wave length of light upon the accelerated weathering of asphalts, each of the five asphalts were exposed behind light filters which cut off various proportions of the ultraviolet region of the spectrum. Three-inch square light filters having "cut-off" points of 2860, 3230, 3550, and 4200 Å were obtained and placed in front of the bottom half of the panels during the test procedure. The transmission characteristics of the four filters are shown in Fig. 2. The transmission curves for these filters are similar, the difference lying in the increasing value of the "cut-off" point below which no light is transmitted.

The effect of these light filters upon the average accelerated weathering of the five asphalts used in the investigation are shown in Fig. 3. These data were obtained during run Nos. 1, 3, and 5, all of which were made at a constant operating temperature of 140 F. but with amperages of 15, 16, and 17, respectively. Figure 3 also shows the value of the oxalic acid decomposition for 17.5 hr. obtained with the cell placed behind the filters. The weathering results obtained confirmed the earlier work done to show the correlation of the amperage with accelerated weathering in that comparable accelerated weathering values were obtained as long as some ultraviolet was left in the spectrum. The values obtained behind filter *D* show very slow weathering coincident

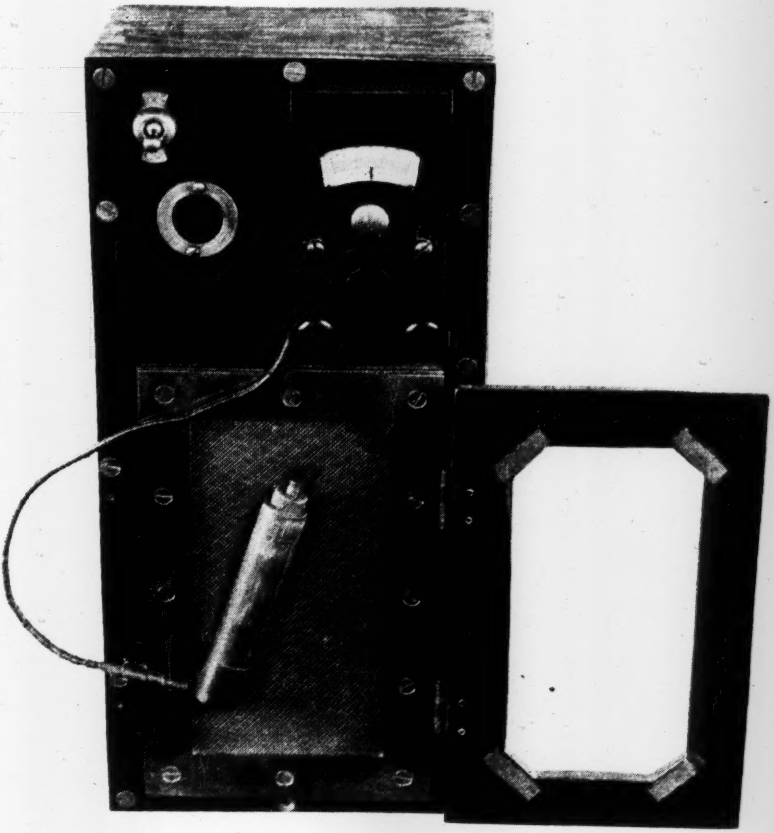

FIG. 4.—Electrometric Failure Detector.

with the elimination of all the ultraviolet rays from the light. In order to guarantee proper light emission, the globe should be cleaned daily.

Failure Detection:

During the first three runs the panels were examined visually for failure by holding them under a 60 w. electric light bulb. The reproducibility of results for the sets of duplicate panels were far from satisfactory when considering the great care with which the panels were made and the runs conducted. Unsatisfactory duplication of results was especially noticeable in the case of those asphalts which fail either by the formation of very fine cracks or "pin points." These types of failures are very difficult, and in some cases impossible, to evaluate accurately by visual means. If a fine crack is not normal to the panel surface, light cannot be reflected back through the crack for observation.

In order to eliminate errors in determining failure by the visual means during run Nos. 4 and 5, the electrometric method suggested by Strieter (2) was used together with the visual method. This electrometric apparatus consisted of two dry cells, a 0.5 megohm resistance, a galvanometer, an electrolyte cell, and the asphalt panel connected in series. A short, round camels hair brush was inserted through the dowell pin in the end of the electrolyte cell to insure more intimate contact of the solution and asphalt panel. This apparatus is contained in a 4 by 5 by 12 in. box for convenient use. A photograph of the apparatus is shown in Fig. 4.

The electrolyte cell contains a 2 per cent solution of magnesium chloride in a 5 per cent alcohol solution. The electrolyte is applied to the asphalt surface through the camels hair brush, and if any cracks extend through the asphalt to the aluminum panel, a galvanometer needle deflection is obtained. All deflections are taken as evidence of metal exposure. Panels are considered as having failed when two deflections are obtained more than $\frac{1}{4}$ in. from the edge of the panel. The template recommended by Strieter (2) was not used as the area of the holes covered only 18.9 per cent of the panel.

Failure of panels during runs Nos. 4 and 5 by the two methods are shown in Table III. The two methods produce identical results on asphalts which fail by the opening of wide cracks. The electrometric method detects failure considerably earlier than the visual method in the case of those asphalts which fail by the opening of either fine cracks or "pin points."

TABLE III.—VISUAL *versus* ELECTROMETRIC METHODS OF DETERMINING PANEL FAILURE.

	Asphalt No. 1	Asphalt No. 2	Asphalt No. 3	Asphalt No. 4	Asphalt No. 5
Type of failure	Wide cracks	Pin points	Very fine cracks	Pin points	Wide cracks
Cycles to failure, run No. 4 (160 F., 16 amp.):					
Visual	41	23	24	22	25
Electrometric	41	21	22	22	25
Run No. 5 (140 F., 17 amp.):					
Visual	65	52	53	46	50
Electrometric	65	45	43	40	51

Refrigeration Temperature:

The effect of the refrigeration temperature on the accelerated weathering results was also investigated as the cracks in the asphalt on the panels result first from thermal contraction during the refrigeration period. Refrigeration temperatures of −20, −10 and +10 F. were used. The accelerated weathering results obtained when using the above refrigeration temperatures are shown in Table IV. These results indicate that the accelerated weathering of asphalts is not affected by refrigeration temperature variations within the range of +10 to −20 F.

Miscellaneous Variables:

During the course of this work experments were made to determine whether other miscellaneous variables such as force of the water spray, surface cleanliness of the aluminum panel, and means of panel support affected the test results.

TABLE IV.—EFFECT OF REFRIGERATION TEMPERATURE ON ACCELERATED WEATHERING RESULTS.

	Asphalt No. 1	Asphalt No. 2	Asphalt No. 3	Asphalt No. 4	Asphalt No. 5	Average
Cycles of Failure						
Run No. 4 (160 F., 16 amp.):						
Refrigeration Temperature, −20 F.	40	25	21	24	23	27
Refrigeration Temperature, −10 F.	44	21	22	21	25	27
Refrigeration Temperature, +10 F.	42	23	21	22	31	28
Run No. 5 (140 F., 17 amp.):						
Refrigeration Temperature, −20 F.	75	43	38	40	49	49
Refrigeration Temperature, −10 F.	66	45	40	42	52	49
Refrigeration Temperature, +10 F.	72	41	37	40	52	48

During the weathering of asphalt films, the surface becomes coated with asphaltenes which might conceivably retard the oxidation rate of asphalt unless they are subsequently removed by the water spray action. Tests were made on duplicate sets of panels, one of which was subjected to the normal water spraying while the second was immersed in a water bath. No difference in results was obtained.

Removing the carbonized oil film, which characterizes aluminum sheet, by strong caustic action followed by acetic acid and water rinsing had no effect on the accelerated weathering test results.

Panels supported on the revolving drum by means of clips (as used in this work) had the same weathering life as those supported by the conventional panel holder supplied with Atlas machines.

SUMMARY

This investigation of the effect of operating variables upon the accelerated weathering of asphalts has shown that the temperature of test must be accurately controlled if comparable results are to be obtained between laboratories. It is recommended that 140 F. black bulb panel temperature be used as the best temperature to obtain values which will adequately differentiate between the weathering characteristics of different asphalts in the shortest practical time.

The electrometric method for detecting failure of panels is essential for accurate results on those asphalts which fail by the formation of fine cracks or "pin points."

Under normal conditions, amperage, refrigeration temperature, force of water washing, and surface cleanliness of the aluminum panels have no effect upon the test results.

Acknowledgment:

The author wishes to extend his appreciation to the laboratory personnel who performed the experimental work and offered constructive criticism of this presentation.

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(3) R. R. Thurston, and E. C. Knowles, "Asphalt and Its Constituents—Oxidation at Service Temperatures," ***Industrial and Engineering Chemistry***, Vol. 33, pp. 320–323 (1941).

DISCUSSION

Mr. G. L. Oliensis.[1]—I have been very much interested in the useful data contained in this paper. I have for many years studied the service behavior of asphalts employed in roofings, and have compared it with the results shown by the weatherometer on the same asphalts.

It seems to me that weatherometer tests are very instructive and helpful, but that a note of caution might be sounded in regard to their interpretation, because there are two rather important factors that are not sufficiently taken into consideration in construing them, and yet which do markedly influence weathering resistance.

One of these factors in roofing exposed to actual weathering is introduced by wind stresses and vibration, which may open up numerous "hair-cracks" in the coating layer, especially during the winter season when the roofing is more or less brittle. These hair-cracks may be so fine as to escape detection with the naked eye, and yet may extend all the way down to the saturated felt and provide open channels through which the destructive elements of weathering may penetrate to attack the felt. No matter how inherently sound a coating may be and how resistant to deterioration, if the vibration or flexion to which the roofing is subjected should crack that coating and thus expose the bare saturated felt to the weather, fairly quick disintegration is quite likely to take place and the question then arises: Of what avail is the use of a high-grade coating if that coating is cracked and its protective function thus impaired?

Unfortunately, there is no way to determine the effect of these wind stresses in the weatherometer tests as presently conducted, because the coatings are placed on rigid aluminum panels and wind or vibration is definitely *not* one of the forces to which they are subjected.

And then there is another factor that is probably of equal importance: In the weatherometer the coatings are applied upon aluminum panels, and yet it is but seldom that these asphalts are applied on metal in actual service. They are applied usually on asphalt saturated felt or other similar fabrics, and I am convinced that there is a big difference in the behavior of asphalts on metal and their behavior on fabrics that have been impregnated with asphalts. It is, of course, much more difficult to evaluate the failure of layers of asphalt upon saturated fabrics in the weatherometer than it is to evaluate it upon metal panels. This is a fairly valid reason for the use of the latter, and yet none the less an important factor is thereby being omitted from the test.

My point in making these remarks is to call attention to the fact that these two factors do markedly affect the showing of asphalt in any weathering tests; that as long as they are omitted from consideration in our weatherometer tests we have not "gotten all of the packing out of the case," so to speak; and they will somehow have to be included in our weatherometer tests before we shall be able to foretell with reasonable accuracy how a certain type of coating or a certain type of roofing will behave in actual service.

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[1] Chemist in Charge, Technical Dept., Barber Asphalt Corp., Madison, Ill.

MR. J. S. MILLER.[2]—In conducting the weatherometer test as carried out by most of us up to the present time, an electrolyte has been used. Specimens that are carried through, we will say 35 or more cycles and tested periodically, generally show some surface action of the electrolyte upon the specimen. The degree of effect of the electrolyte is an unknown factor, and I am sorry that in this paper the author has not given us some other means of detecting incipient cracking or failure other than by the use of an electrolyte.

When I was with the Barber Asphalt Corp., we used a spark gap for quite a while, which obviated the use of an electrolyte, and I believe that method is equally as efficient in determining the incipient cracking as is the conductivity through means of an electrolyte. I am wondering whether it would have detected fine cracks earlier or later than the author reports.

If he has done any work along the lines of studying other means of detecting failure, it would be very helpful to include description of the methods even though they were not thought satisfactory.

MR. M. R. BEASLEY.[3]—I wonder, in connection with this work, whether the author made any effort to compare weatherometer tests with duplicate panels exposed to actual weather.

In our work on accelerated weathering, which started in 1925, we found that pure weatherometer tests are not to be relied upon. They must be compared and correlated with duplicate panels exposed to natural weather in climate under investigation, in order to gather the necessary experience so that later accelerated tests may be depended upon.

I should also like to take issue regarding the purely mechanical examination of failure. We realize that it would seem that the value of such a mechanical examination lies in the fact that the mechanical tests make it easier for different operators to correlate their results. On the other hand, we have found that accelerated weathering of asphalt roofing and roofing materials is not a test that can be conducted by the uninitiated. We have found that the visual examination based on some experience with natural weathering conditions is necessary in that there are many factors which are shown up in accelerated weathering which are not entirely shown up by a mechanical test, such as the spark or the electrolyte method. For instance, asphalt films do crack, but frequently the asphalts that first show hairline cracking in accelerated tests are not always the first asphalts to go to pieces on a roof. Some asphalts may also fail prematurely in accelerated tests due to excessive mounding, which in turn is caused by excessive temperatures.

I should like to ask the author whether he has done anything towards correlating these results with material exposed to natural weather.

MR. A. HOLMES.[4]—We have worked with the weatherometer along the same lines as the author has described in an attempt to obtain a correlation between the results between outdoor weathering and the weatherometer. The panels were exposed in both the South—in Louisiana—and in New Jersey. Our final conclusion after several years' work was that the weatherometer can serve to distinguish a very good asphalt from a very bad one but is of little use for grading asphalts between these extremes.

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[2] Asphalt Technologist, Rahway, N. J.

[3] Superintendent, Eastern Roofing Div., Bird & Son, Inc., East Walpole, Mass.

[4] Chemist, Process Div., Esso Laboratories, Standard Oil Development Co., Elizabeth, N. J.

MR. V. A. ENDERSBY.[5]—Some years ago panels we exposed in various locations from Seattle to New Orleans, and found the correlation very poor between those panels and the weatherometer. After seeing this paper, I have concluded that a large part of that was due to exactly what is pointed out in the paper.

MR. BRUCE WEETMAN[6] (*author's closure, by letter*).—A limited amount of work was done with an available spark-gap instrument designed for detecting leaks in vacuum apparatus. When used on accelerated weathering panels, sparks were produced not only at locations of cracks, but over areas which definitely contained no cracks. The spark intensity could not be adjusted satisfactorily so that sparking occurred only at the cracks and the method was abandoned.

We made no attempt to correlate these accelerated weathering results with outdoor weathering. This investigation was undertaken solely for the purpose of determining what limitations would have to be placed on the operating variables of the accelerated test to enable different laboratories to obtain comparable results. To be of maximum value, the accelerated weathering test should be correlated with outdoor weathering. This would have to be done on an extensive scale to insure the success of the work.

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[5] Research Engineer, Shell Development Co., Emeryville, Calif.

[6] The Texas Company, Beacon, N. Y.

A METHOD FOR THE TESTING AND EVALUATION OF ROAD TARS

By E. O. Rhodes[1] and H. E. Gillander[2]

Synopsis

Road tar specifications of the type most frequently used give satisfactory control over quality in the great majority of instances. However, the significance of some of the tests is open to question and it would be desirable to have tests that could be interpreted directly in terms of road performance. This problem has been attacked by a number of investigators.

The authors report on an accelerated weathering procedure that, when applied to a series of road tars, gave results that agreed with their observed performance in service. The test procedure makes it possible to bring about extensive alterations in some tars in one day's time. It gives (*a*) an estimate of the amount of evaporation that can be expected from the tar, (*b*) its probable rate of set up and ability to develop proper stability, and (*c*) its tendency towards embrittlement. This test procedure is a further development based on work done with an accelerated weathering procedure reported in 1936.

Data are presented indicating that in general, and particularly in the case of hot application grades of road tar, the softening point of the 300 C. distillation residue can be used to estimate the probable durability. Other data indicate that the present distillation limits on some grades of cold application road tars are too wide to control the quality adequately.

Road tar specifications have been considerably modified and improved in recent years. Originally, the specifications included only simple tests that gave some hint of the source of the material. As new types of tars became available and as new construction practices came into use the specifications were altered or expanded when it was found that control was inadequate. The present specifications offer control over quality, partly by limiting certain controlling factors and partly by restricting the source of the crude tar used to make the road tar.

While this type of specification has served satisfactorily in the great majority of instances, it has been realized that there should be more information on the practical significance of the various tests employed. Furthermore, it would be desirable to have tests which would be capable of direct interpretation in terms of road performance. A specification built up on such a basis would tell whether the tar had the proper consistency required for the type of construction being employed, whether it would adhere to the aggregate, how it would set up, and would give some indication of its probable durability.

Present specifications appear to cover

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[2] Tar and Chemical Division, Koppers Co., Pittsburgh, Pa.

the consistency requirements adequately. In the case of adhesion, a number of tests have been developed, and of these the Nicholsen (**1**)[3] test appears well adapted from the standpoint of practicability and laboratory technique. A considerable amount of work that could lead to estimates of the durability of road tars has been carried out, but most of it has been confined to correlations of the present test methods with weathering—either natural or simulated. The Public Roads Administration has reported (**2, 3**) on work where the relation between the specification tests and the weathering process was investigated. The British Road Tar Association has reported (**4**) an extended investigation wherein a series of tars of RT-10 consistency was used in premix construction in a number of localities and also subjected to new as well as the usual laboratory tests. The British investigators found no general relation between the test data and service results. They were able to develop from the data a relationship between a modified softening point of the 300 C. distillation residue and service performance by applying an oxidation factor having a magnitude determined by statistical analysis. If their data are examined, however, it is seen that the tars fall into two groups. In the case of the true high-temperature tars, coke-oven and horizontal retort tars, the unmodified softening point of the 300 C. distillation residue can be correlated with the service performance of the tars, the vertical retort tars showing a different relationship. While it is known that tars do oxidize, the known differences in character of the tar resins (C-2) of vertical retort tars and high-temperature tars (**5**) make it possible that the separate grouping should occur and the application of an oxidation factor may not be justified. It should be pointed out that vertical retort tar is produced to only a very minor extent in the United States.

The causes of the aging of tars have been the subject of a number of investigations. In those cases where attempts were made to evaluate the importance of the weathering factors, it was agreed generally that tars change primarily as the result of evaporation of lower-boiling oils and to a lesser extent because of oxidation (**6**).

DEVELOPMENT OF ACCELERATED WEATHERING METHOD

On the basis of the available knowledge of the weathering process, the authors devised and reported (**6**) on an accelerated weathering method for road tars. The method consisted in preparing a mix of 3 per cent by weight of the road tar and 97 per cent by weight of 20 to 30 mesh Ottawa silica sand. This mix was placed on a rotating pan in an oven held at 60 C. At intervals, portions of the mix were removed and tested for (*a*) percentage loss by evaporation, (*b*) stability by a modified Hubbard-Fields test on a 15-g. compressed specimen employing a plunger smaller than the orifice to obtain a truer shearing action, and (*c*) degree of brittleness by a sand-blast test wherein a 15-g. compressed specimen was blasted with 50 g. of 20 to 30 mesh Ottawa silica sand at 10 lb. air pressure.

This method proved to be of considerable value in testing road tars and formulating blending procedures. The method had obvious deficiencies, however, and as more extensive data were accumulated it became apparent that it was not always reliable in the case of cold-application grades of road tars.

In 1939 an opportunity was presented to investigate four different types of road tar which were found to be giving varying results in service. These tars

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[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1174.

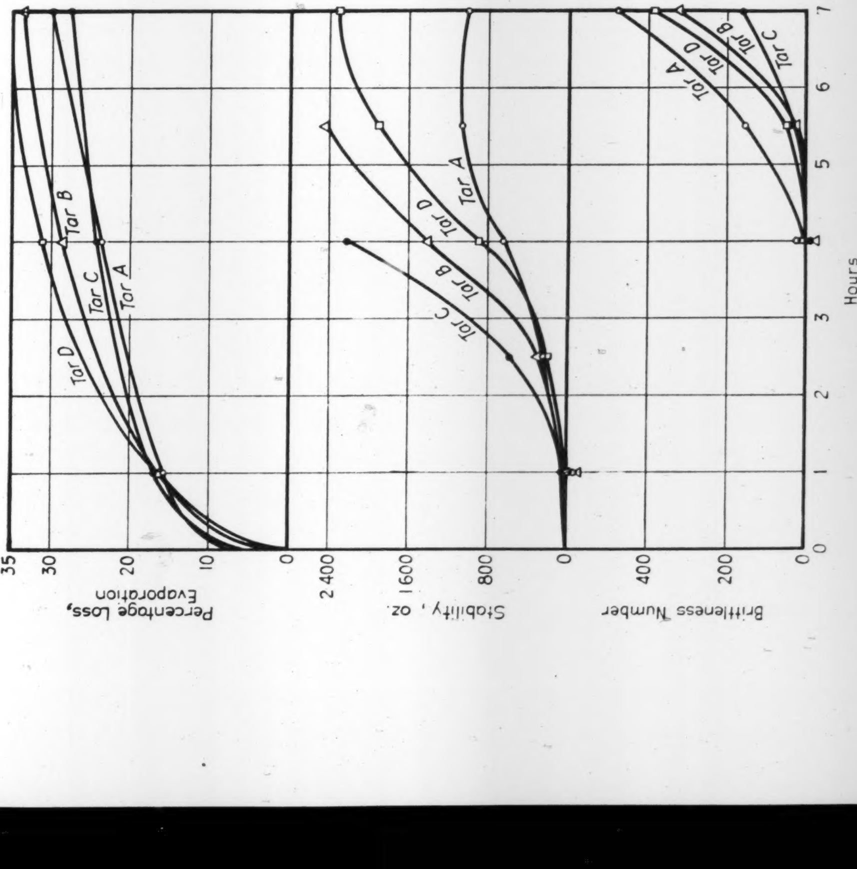

FIG. 1.—RT-3 Grade Road Tars, Accelerated Weathering at 100 C.

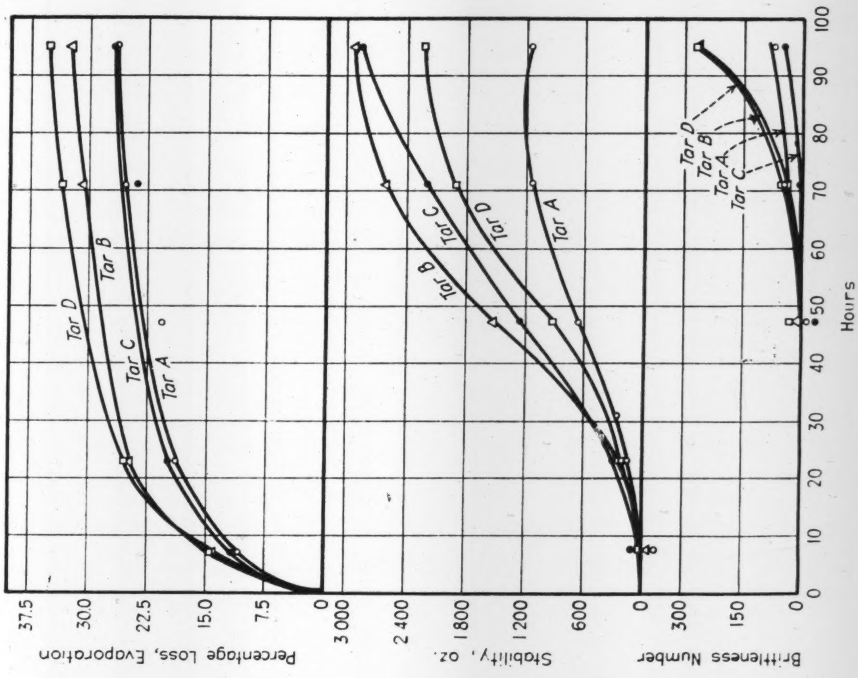

FIG. 2.—RT-3 Grade Road Tars, Accelerated Weathering at 60 C.

were applied during the same period on a single type of construction in one district. As identified in Table I, tar C was completely satisfactory and tar A was giving passable results. On the other hand, roads treated with tar D were ravelling extensively within a few months and treatments with tar B were exhibiting incipient ravelling in that time.

TABLE I.—RT-3 GRADE ROAD TARS USED IN ACCELERATED WEATHERING TESTS.

	Tar A	Tar B	Tar C	Tar D
Specific gravity at 25°/25°C.	1.10	1.12	1.17	1.10
Specific viscosity, Engler, 40 C.	17.7	14.8	20.8	17.2
Distillation test on water-free material—total distillate by weight, per cent:				
to 170 C.	0.4	1.3	1.6	1.3
to 235 C.	8.6	11.2	9.2	7.7
to 270 C.	20.7	23.8	19.3	26.4
to 300 C.	28.7	33.9	26.4	35.7
to 355 C.	48.3	52.6	41.9	52.8
Softening point (ring-and-ball method) of residue from distillation tests, deg. Cent.:				
300 C. residue	40.5	53.4	38.7	47.4
355 C. residue	77.5	113.5	74.4	101.8
Tests on distillates:				
(*a*) Specific gravity at 38°/38°C.:				
0 to 300 C.	0.963	0.979	1.023	0.939
300 to 355 C.	1.029	1.050	1.100	1.020
(*b*) Sulfonation index:[a]				
0 to 300 C.	4.6	1.8	0.6	3.0
300 to 355 C.	1.8	0.5	0.0	1.4
(*c*) Surface tension,[a] dynes per cm.:				
0 to 300 C. (30 C.)	34.1	37.7	41.5	35.2
300 to 355 C. (50 C.)	35.0	37.0	42.4	36.5

[a] Sulfonation Index determined by producer's method (5).

Surface tension determined by Du Noüy tensiometer.

All other tests made in accordance with A.S.T.M. Tentative Specifications for Tar (D 490 – 38 T), 1942 Book of A.S.T.M. Standards, Part II, p. 1247.

The tars were subjected to accelerated weathering by the method just described, first at 100 C. to obtain results more promptly and then at the usual 60 C. temperature. The results of these tests are shown in Figs. 1 and 2. The tests run at 100 C. provided little information, because no real differentiation was obtained in degree of brittleness, and tar A which was giving passable service results actually appeared to be the poorest. The tests run at 60 C. aligned the tars in the order of serviceability found on the roads, but here also the amount of differentiation was not sufficiently great.

Examination of the data shows that in the two tests tars B and D maintained their relative positions for rate of increase in stability. However, dropping the test temperature from 100 C. to 60 C. caused the stability curves for tars A and C to change relative to those for B and D in the direction necessary to provide closer agreement with the field results. The lower test temperature also differentiated the tars more accurately in degree of brittleness. This indicated that a test temperature lower than 60 C. might provide still better correlation.

The results obtained for tar A also indicated the necessity of a lower test temperature. This tar was one of the few vertical retort tar blends being produced commercially in this country. Adam, Shannon, and Sach (5) have shown that upon atmospheric exposure at ordinary temperatures low-aromatic (vertical retort) tar changes more slowly than high-aromatic (coke-oven) tar, both in degree of evaporation and viscosity, while at higher temperatures the difference is no longer apparent. In addition, Adam, Murdoch, and Mott (7) have made tests which indicate the transition temperature for this change in character of the vertical retort tar to be 32 C. On this basis an accelerated weathering test should be run at a temperature lower than 32 C. if the true character of a vertical retort tar blend is to be revealed.

It was found that no reasonable degree of acceleration could be obtained in testing the tar-sand mixes in the oven-type weatherometer at temperatures lower than 60 C. Accordingly, a new procedure for accelerated weathering was devised wherein air is forced through the tar-sand mix contained in tubes. Two ±0.05 per cent by weight of the tar is mixed with 98 per cent by weight of 20 to 30 mesh Ottawa silica sand. The amount of tar in the mix is checked by igniting three samples over a Bunsen burner, the loss in weight being taken as the amount of tar. One hundred gram portions of the mix then are packed into aluminum tubes 9 in. long and 1 in. in diameter, the mix being held in place on the bottom by cloth gauze fastened with a rubber band and on the top by a steel wool plug about ¼ in. thick. The mix is placed in the tube in several portions, being compacted uniformly by light tamping with a wood plunger after each addition. This assembly (less than 200 g.) is weighed on an analytical balance to the nearest milligram. Weighings before and after weathering give the loss by evaporation of the tar and the percentage loss is calculated on the basis of the known percentage of tar in the mix. A separate tube is prepared for each time interval that is to be represented in the weathering test, five tubes for 1, 3, 7, 16, and 23-hr. tests being used generally when it is desired to obtain the contour of the curves representing the rates of change. Accelerated weathering is carried out in the apparatus shown in Fig. 3. The tubes are placed in the weathering chamber which is a steel box completely insulated. Filtered and oil-free air at a pressure of about 0.8 psi. is furnished by a rotary type blower. The air passes first through a copper coil immersed in a conditioning bath *G* which is equipped with both heating and cooling appliances. The

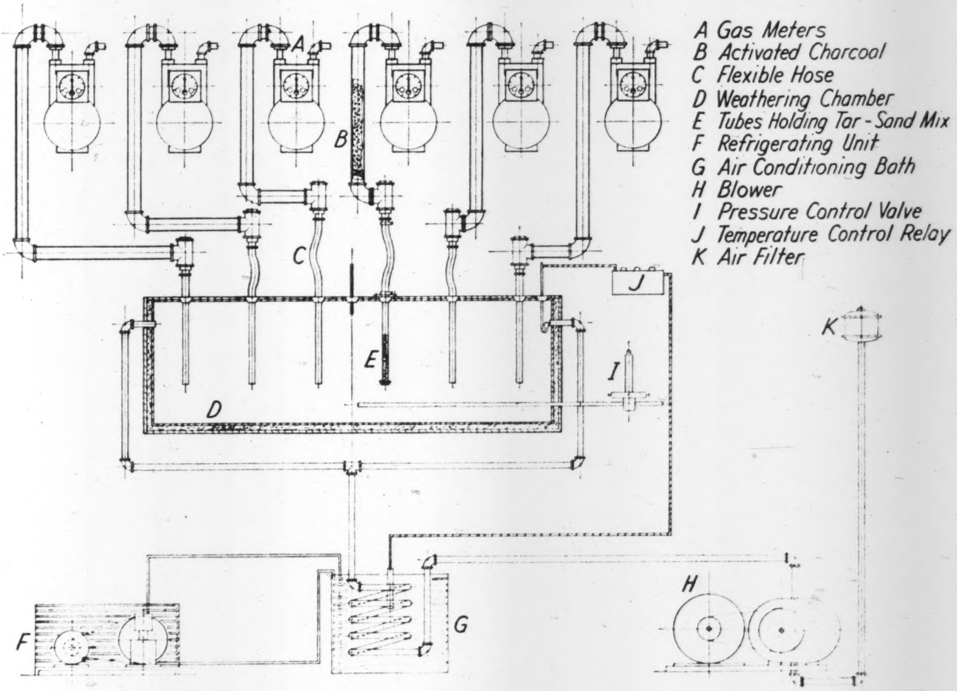
A Gas Meters
B Activated Charcoal
C Flexible Hose
D Weathering Chamber
E Tubes Holding Tar-Sand Mix
F Refrigerating Unit
G Air Conditioning Bath
H Blower
I Pressure Control Valve
J Temperature Control Relay
K Air Filter
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Fig. 3.—Accelerated Weathering Apparatus.

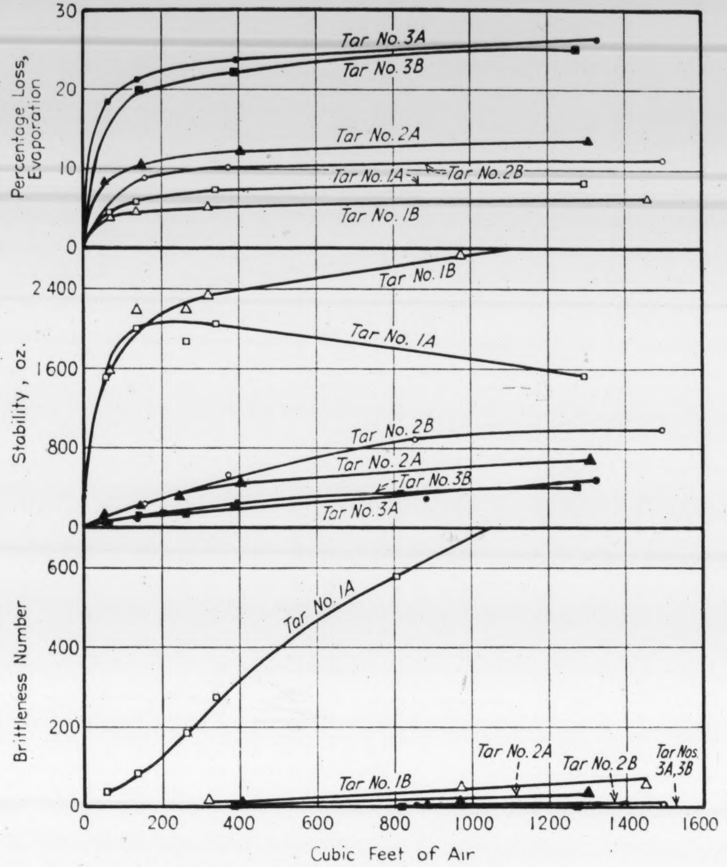

FIG. 4.—Accelerated Weathering at 30 C.

Tests showing the effects of amount of tar in mix. Curves marked "A" represent mixes containing 3.0 per cent tar, B curves, mixes containing 2.0 per cent tar.

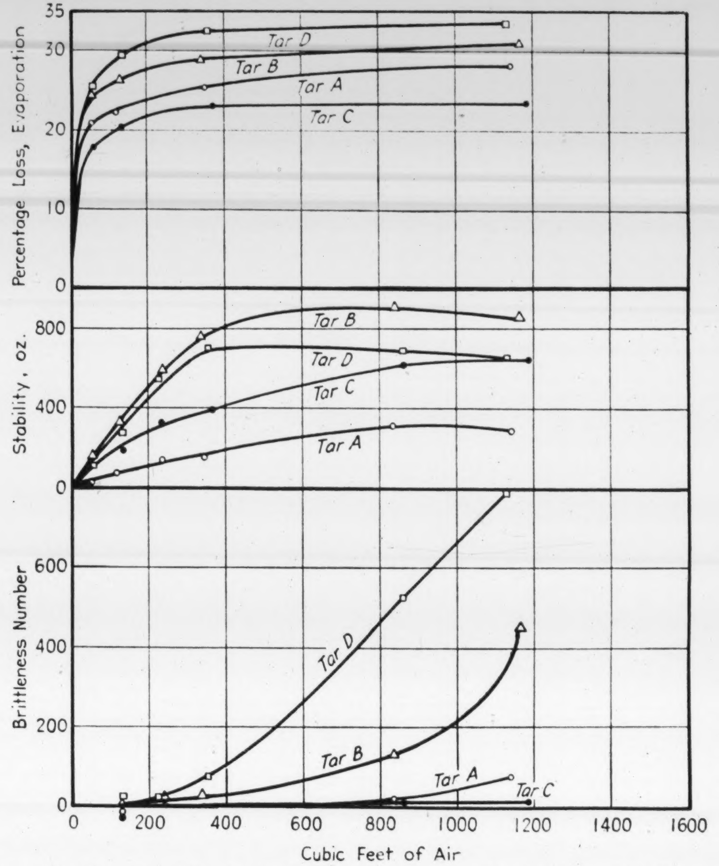

FIG. 5.—RT-3 Grade Road Tars, Accelerated Weathering at 30 C.

temperature of this bath is controlled by a thermoregulator located in the weathering chamber in front of one of the two air inlets. This regulator adjusts the temperature of the conditioning bath so that the air enters the weathering chamber at a temperature of 30 ± 0.1 C. The blower *H* supplies over 14 cu. ft. of air per minute and this volume is sufficient to maintain the insulated chamber at the required temperature. The air passes up through the mix at a rate of 50 to 60 cu. ft. per hour and the volume passing through each tube is measured by an individual meter. The rate is controlled by means of an adjustable air pressure regulator *I* on the excess air outlet. At the proper time interval, a tube is removed from the chamber and reweighed to determine the amount of evaporation of tar oils that has taken place. The tar-sand mix then is removed from the tube and mixed thoroughly, separating the mass into discrete particles if the tar has become sufficiently brittle or "dead." As in the previous work (6), 15-g. portions of the mix are weighed into cylindrical molds 1 in. in inside diameter, held in a 25 C. oven for 30 min., subjected to a pressure of 3300 psi. for 1 min., and returned to the 25 C. oven for 10 min. Three specimens are tested for stability and two for degree of brittleness using an air pressure of 8.5 psi., the average being taken in each case.

This change in test procedure gave satisfactory acceleration of the weathering process at 30 C., making it possible to bring about extensive alterations in some tars in a period of one day. In addition, the new procedure caused the stability of the tar-sand mix to change in the expected manner. In the case of accelerated weathering carried out in the oven-type weatherometer, rate of increase in stability lagged at the start of the test. It was realized that this action did not represent service behavior because a normal road tar should exhibit a rapid increase in stability at the start of the test when the volatile oils are evaporating, the rate decreasing slowly thereafter. The new test procedure causes the stability to change in the proper manner.

The new procedure brought about the desired acceleration of weathering, but mixes containing 3 per cent of tar were not differentiated to the degree that should be expected. It was found, however, that decreasing the tar content of the mix from 3 to 2 per cent altered the weathering characteristics of tars to different degrees. Figure 4 gives the results of accelerated weathering on three tars tested using, first, mixes containing 3 per cent tar, and then mixes containing 2 per cent tar. It is seen that tars Nos. 2 and 3, which are known to be durable road tars, did not alter in essential character. Tar No. 1, a binder which experience has shown to be of poor character except under optimum conditions, gave widely different results in regard to change in stability and degree of brittleness in the two tests, the 2 per cent mix giving results in the expected order.

DISCUSSION OF RESULTS

Figure 5 gives the results obtained on tars A, B, C, and D (Table I) plotted against the number of cubic feet of air passed through the mix. Tar A, the vertical retort tar blend, increased in stability very slowly in a manner corresponding to the known slow-setting characteristics of this type of tar. Towards the end of the test it became somewhat "dead," as shown by the slight decrease in stability, but it did not become excessively brittle. Tar B was next to the poorest in service and it underwent drastic changes in the weathering test, increasing rapidly in stability to a maximum and dropping off thereafter, while brittleness appeared at an early age and became more severe as weathering progressed. Tar C, which gave completely satisfactory results in service, increased in stability quite rapidly at the start of the test and more slowly thereafter; but it was only slightly brittle at the end of the test. Tar D, which gave the poorest results in service, also was the poorest by this test, changing even more extensively than tar B.

TABLE II.—PENETRATION GRADE ROAD TARS USED IN ACCELERATED WEATHERING TEST.

	Tar No. 1	Tar No. 2	Tar No. 3	Tar No. 4
Specific gravity at 25°/25° C.	1.194	1.195	1.192	1.198
Float test, at 50 C., sec.	174	170	175	142
Total bitumen, soluble in carbon disulfide, by weight, per cent	91.7	91.7	92.7	92.3
Distillation test on water-free material—total distillate by weight, per cent				
to 170 C.	0.0	0.0	0.0	0.0
to 200 C.	0.3	0.0	0.0	0.0
to 235 C.	0.9	0.2	0.1	0.0
to 270 C.	5.8	3.5	3.5	1.5
to 300 C.	13.6	10.8	10.7	6.9
to 355 C.	31.0	27.6	29.9	27.4
Softening point (ring-and-ball method) of residue from distillation tests, deg. Cent.:				
300 C. residue	53.5	49.0	46.0	39.7
355 C. residue	92.5	80.0	81.5	70.0
Specific gravity of distillates at 38°/38° C.:				
0 to 300 C.	1.008	1.010	1.013	1.019
300 to 355 C.	1.071	1.067	1.071	1.078

NOTE.—Tests made in accordance with A.S.T.M. Designation D 490 - 38 T.

The rate of evaporation curves show that tars B and D lost considerably more oils than either tars A or C. This excessive loss of oil is one reason for the poor performance of these tars because such an action naturally would result in a thinner film of residual binder being left on the aggregate after the mix had set up. Referring again to Table I, it will be seen that tars B and D had excessive amounts of distillate boiling below 270 C. and 300 C. It is interesting to note that a number of state highway departments have recognized the need for closer control on the distillation limits and now include maxima that tars B and D could not meet.

Mention has been made of the English work on a "transition temperature" governing the characteristics of vertical retort tars. The vertical blend tested in this series of tars did not exhibit any such phenomenon, even in the rate of evaporation characteristic which should be affected the least by the variations in weathering procedure.

Table II and Fig. 6 show data obtained on a series of penetration grade road tars made from the same type of crude tar. These data show that as the softening point of the 300 C. distillation residue becomes lower, the tars become less susceptible to embrittlement and that this softening point can serve as an indication of the durability of the road tar.

The curves showing the rates of evaporation of these tars illustrate a tendency which has been noticed repeatedly in the case of higher consistency materials made from various types of crude tar. The percentage of evaporation after the passage of 100 cu. ft. of air is in the expected order when judged by the distillation data. At the end of the test the order of evaporation has been reversed so that tar No. 1, which had the largest amount of distillate boiling below 300 C., has lost the smallest amount of oil by evaporation. This action might be explained on the basis of oxidation which would tend to increase the weight and counteract the loss in weight coming as the result of evaporation. However, these four blends are combinations of the same

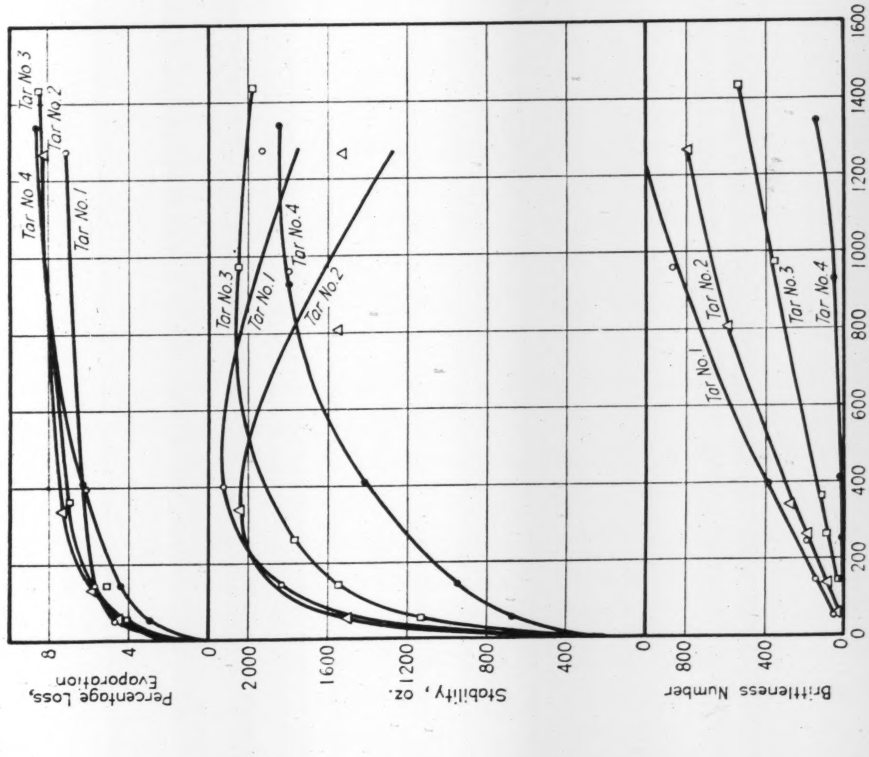

FIG. 6.—Penetration Grade Road Tars, Accelerated Weathering at 30 C.

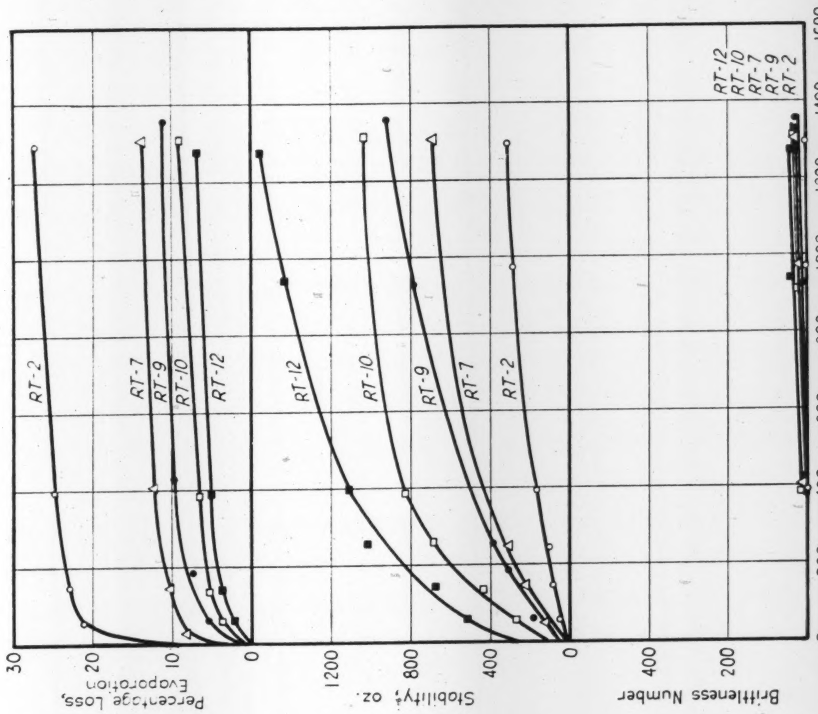

FIG. 7.—Road Tars Made from One Base Tar, Accelerated Weathering at 30 C.

crude tar with relatively small amounts of high-boiling oil and it does not appear likely that the variations in composition could result in such an oxidative effect. It is possible that the action may be the result of an alteration in the structure of the tar so that diffusion of volatile constituents to the tar-air interface is hindered.

The curves for tars Nos. 1, 2, and 3 giving the rate of change in stability all reach a maximum and then decrease. This action comes from progressive loss of plasticity as weathering proceeds and mixes weathered 23 hr. could not be molded into as strong test specimens as those weathered for only 7 hr., the result being a lower stability as well as a higher brittleness number. On the basis of previous work (6) it is believed that if sand mixes of these tars were molded and weathered in specimen form the stability would continue to increase over a very extended weathering period.

Figure 7 illustrates the effect of accelerated weathering on a series of road tars of different consistencies made from the same base tar. They are given to show the amount of evaporation and relative degrees of stability that might be expected from the different grades of road tar. The amount of evaporation possible varies according to the grade in the expected order. The higher consistency grades develop a much higher stability, partly because of the greater amount of distillation required to reach the proper consistency and partly because of the thicker films in their tar-sand mixes. Since the base tar was of a durable type none of the tars became brittle to any appreciable extent.

CONCLUSION

A test method has been developed for evaluating the weathering properties of road tars that, in the case of a series of tars, gave results agreeing with their service behavior. Air at 30 C. is passed through a tar-sand mix having tar films in the order of 0.006 mm. thick and extensive weathering is brought about in 23 hr. time. The test simulates the action that takes place on the surface of a densely graded or tightly sealed road but also will represent the action in the interior of an open-graded road. The test results, accordingly, must be interpreted on the basis of knowledge of the road design.

The test has been used to some extent in studying road tar specifications. The results indicate that, in general, and particularly in the case of the hot application grades, the softening point of the 300 C. distillation residue may be used to estimate the serviceability of the road tar when employed with proper types of construction. They also indicate that it is desirable to have closer distillation limits in the case of the cold application grade road tars.

The method in its present form is suitable for testing those materials which change primarily as the result of evaporation. The acceleration is obtained as the result of using extremely thin films which permit, practically speaking, the maximum amount of evaporation in a given time. The method cannot be used to evaluate materials which change through oxidative processes which may or may not be accelerated by light. Work with this test is being continued.

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THE GIESELER METHOD FOR MEASUREMENT OF THE PLASTIC CHARACTERISTICS OF COAL

By Glenn C. Soth[1] and Charles C. Russell[1]

Synopsis

A modified Gieseler plastometer together with a procedure of operation is described, and a method of calibrating the apparatus, to express the results of tests in absolute units, is given. The problems met in the measurement of plastic characteristics of coking coals and the limitations in the use of absolute units to express the measurements are discussed.

Typical results are given for measurements of the plastic characteristics of different ranks of coal and the limits of reproducibility of results are shown. Generally, significant temperature points may be duplicated within 5 C., and maximum fluidity, expressed in divisions per minute, may be duplicated within 10 to 15 per cent.

The plastic temperature range of usual coking coals is shown to be between 340 and 510 C. when the coals are heated at a rate of 3 C. per min. The range of plastic properties measured by this modified Gieseler plastometer is shown to be equivalent to viscosities between 6.4×10^7 and 6.6×10^2 poises. By changing the load acting to turn the stirrer of the plastometer, this range may be increased.

When coking coals are heated in the absence of air, they soften and fuse. Due to the fact that coals differ in composition, the degree of softening varies. These facts are known to coal technologists and coke-oven operators, and it is believed that variations in the degree of softening are accountable, at least in part, for the character of the coke produced. A very large number of investigations of the softening and fusion of coal have been reported by coal technologists throughout the entire world. Instruments and apparatus of a great variety of design have been described together with results obtained. There is probably no other subject in the realm of coal technology about which there has been so much discussion and controversy.

Coal is a complex heterogeneous material. The organic portion is composed chiefly of carbon, hydrogen, and oxygen, together with smaller amounts of sulfur and nitrogen. Some inorganic substances are always present also, either intimately mixed with the organic material or in the form of free slate. The organic material is seldom homogeneous in its physical make up. Macroscopic constituents have been classified in broad categories either by differences in appearance or by microscopic examination. These different kinds of coal substances are present in different amounts in each coal, some of them being fusible and others infusible.

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It is difficult to define the term "coking coal" because the properties of coals vary according to the conditions of their use. For the purposes of this paper, coking coals may be considered to include all coals which show flow characteristics when they are heated in the absence of air at heating rates ordinarily obtained in coke ovens.

The problem of measuring the plastic characteristics of coking coals is, therefore, quite different from usual plastometric or viscometric work. The measurement of the consistency of tars, pitches, oils, etc., involves essentially a "uniformly heterogenous" system if not a single-phase system. Properties of these materials are relatively permanent and changes occurring in the system during measurement are generally reversible. Measurements of plastic properties of coking coals are made upon a heterogeneous system whose plastic characteristics are transient and, for the most part, changes occurring in the system are irreversible.

The complex nature of the system that exists when coking coals are heated and begin to flow may be shown by a description of the changes that take place at various temperature levels. At room temperature no plastic properties are evident. As coal is heated from room temperature, the first change evident is evolution of water which begins around 100 C. and continues as the coal is heated to higher temperatures. This water may be either water on the surface of the coal particles, water of hydration of some of the compounds in the coal, or water of decomposition formed during carbonization. As the temperature rises above 100 C., molecular rearrangements occur and decomposition begins. At some temperature between 330 and 440 C. most coking coals begin to soften. Rapid decomposition begins around 425 C., producing tar and oil and permanent gases as volatile products. With further increase in temperature, both the degree of fluidity and the rate of decomposition increase rapidly. The fused coal exhibits maximum fluidity generally in the temperature range 440 to 480 C., and the fluidity decreases, usually rapidly, with further increase in temperature until the material resolidifies around 500 C. to semicoke. The rate of evolution of volatile products usually reaches a maximum also in the temperature range 440 to 480 C., and then decreases. Evolution of condensible volatile products usually ceases around 500 C., but the production of fixed gases continues. As the semicoke is heated to temperatures above 500 C., the principal change occurring is further decomposition of the semicoke to form high-temperature coke with evolution of CO, H_2, and some elementary hydrocarbons, such as CH_4 and C_2H_4.

This description indicates that during the time the plastic properties can be measured, active decomposition is occurring. Measurements of plastic properties are not made on a single liquid phase. Instead, measurements are conducted on a heterogeneous system consisting of some solid infusible material, some fluid coal substance, and some gaseous matter present as bubbles in the fused coal. Often the decomposition of the coal and formation of gaseous volatile products cause the melt to swell to several times the volume of the original coal; sometimes the swelling is negligible.

The use of the term "plasticity" and reference to the "plastic properties" of coking coals has been the subject of much argument. There is no accepted terminology to describe the flow characteristics of heated coal. The foregoing description of some of the phenomena involved indicates that the measurement of the flow characteristics of heated coal is a complex problem.

In different publications on this subject, various terms which have specific meaning for rheologists are used. Often terms which possess different meanings from the rheological point of view are used interchangeably by coal chemists. In this paper the term "plasticity" together with the reciprocal term "fluidity" have been used to describe the coal system when it is in a state to exhibit flow characteristics. The term "maximum fluidity" has been used to describe the system when the flow characteristics appear to be present to the greatest degree. These terms appear to describe the coal system satisfactorily until further information will permit more exact definition of the system from the rheological standpoint.

Purpose of the Work:

This work is directed toward establishing a scientific basis for the measurement of the plastic properties of coking coals. Work in this field heretofore has been on an empirical basis and little work has been done to obtain results other than attempts to estimate the relative plastic properties of coking coals.

APPARATUS

The types of apparatus designed by various investigators to study the plastic properties of coal have involved a number of different principles. It will be impossible to describe any of the great number of types of apparatus in detail, but a brief description will be presented of the general schemes used in a few of the instruments that have received the most attention in the United States. An excellent review of the work in this field has recently been published (**1**).[2]

The Foxwell type apparatus was developed by Foxwell (**2**) in England and modified by Layng and Hathorne (**3**) in this country. This apparatus evaluates the plastic properties of coal by measuring the resistance to the flow of nitrogen through a column of coal particles heated at a uniform rate. As the coal softens, there is an alteration in the interstitial spaces through which the gas flows. Plotting the pressure of the nitrogen required to maintain a uniform rate of flow through the mass of coal against the temperature of the coal indicates the extent of softening or plasticity. Importance is also given to the maximum value of the nitrogen pressure. This apparatus has been used extensively in the United States and a large amount of data was accumulated by various investigators before other types of apparatus were designed.

The Agde-Damm dilatometer (**4**) is an instrument developed by the U. S. Bureau of Mines and it is based on the work of Damm (**5**) and Agde and Lyncker (**6**) in Europe. It consists of a tube for holding the coal sample that can be heated at a uniform rate. The coal sample is packed into the tube and a plunger of known weight is placed on the coal. The change in the vertical position of the plunger in relation to the temperature varies with different coals and represents the change in volume of the sample. Actually, the apparatus does not measure a rheological property but the change in position of the plunger indicates that flow characteristics are present over a range of temperature. The changes in volume shown are shrinkage of the sample when the coal softens and the coal particles flow together, and a swelling of the sample when the coal decomposes and bubbles of volatile products form in the plastic mass.

Several penetrometer methods (**1**, p. 64) have been developed in Europe but none of them have been widely used in the United States. These methods in general consist of measuring the depth and velocity of penetration of a weighted wire loop or a needle in a sample of packed, heated coal.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1189.

The Davis plastometer (7), developed by J. D. Davis of the U. S. Bureau of Mines, was one of the first instruments designed to make direct measurement of the plastic flow of coal. The apparatus consists of a small horizontal drum for holding the coal, the drum being rotated inside a furnace that can be heated at a uniform rate. Rabble arms within the drum are connected through a shaft to a device for measuring the torque on the rabble arms and shaft. The sample of coal to be tested, sized between 0 and 20 mesh, is placed loosely in the drum. When the temperature at which the coal softens is attained, resistance develops as is shown by the torque-measuring device. As the coal becomes more fluid with rise in temperature, the resistance drops. Finally, as the coal begins to solidify, the resistance rises again while the semicoke is setting to a solid. Thereafter the resistance drops as the rabble arms are stirring the pieces of semicoke. The principal disadvantage of the Davis plastometer is that it does not evaluate the degree of maximum fluidity attained by the coal.

Methods for the determination of plastic properties using extrusion of softened coal through an orifice as a principle have been studied by Porter (8) and Lum and Curtis (9). A number of difficulties in the design and use of this type of apparatus appear to have prevented further development.

The Gieseler type plastometer operates on a principle similar to the Stormer viscometer. It was originally developed by Gieseler (10) in Germany but his paper did not include sufficient dimensions to permit reproduction of the apparatus. The U. S. Bureau of Mines (11) designed an apparatus based on Gieseler's description which has been called the Modified Gieseler Plastometer. Macura also constructed a plastometer on this same principle and this apparatus is described completely and illustrated by him (12). Because the apparatus designed by the Bureau of Mines was so simple, because it seemed to offer the most promise of yielding results which could be expressed in absolute units, and because it measured the degree of maximum fluidity attained by the coal, this type of apparatus was selected by Koppers Co., Research Department, for studies of the plastic properties of coal.

The Modified Gieseler Type Plastometer:

Since no Bureau of Mines publication contains a description of the plastometer which includes the most recent changes in the design, the present design of the apparatus is described below.

The Gieseler type plastometer is designed so that a sample of coal is packed around a stirring shaft in a retort tightly enough to prevent turning of the shaft until the coal softens. Figure 1 shows a diagram of the assembled apparatus, including the furnace and other auxiliary equipment. Figure 2 is the plastometer itself showing the retort, the plastometer head, and the dial for indicating stirrer movement. Figure 3 shows the different parts of the retort proper which includes the base or crucible, washer, stirrer, and the barrel, and the packing device for placing a sample in the retort for a test. Figure 4 is a cross-section of the retort. The retort crucible or base is of steel, $\frac{27}{32}$ in. in inside diameter and $\frac{5}{8}$ in. in depth and holds the coal to be tested. The center of the crucible bottom has a small

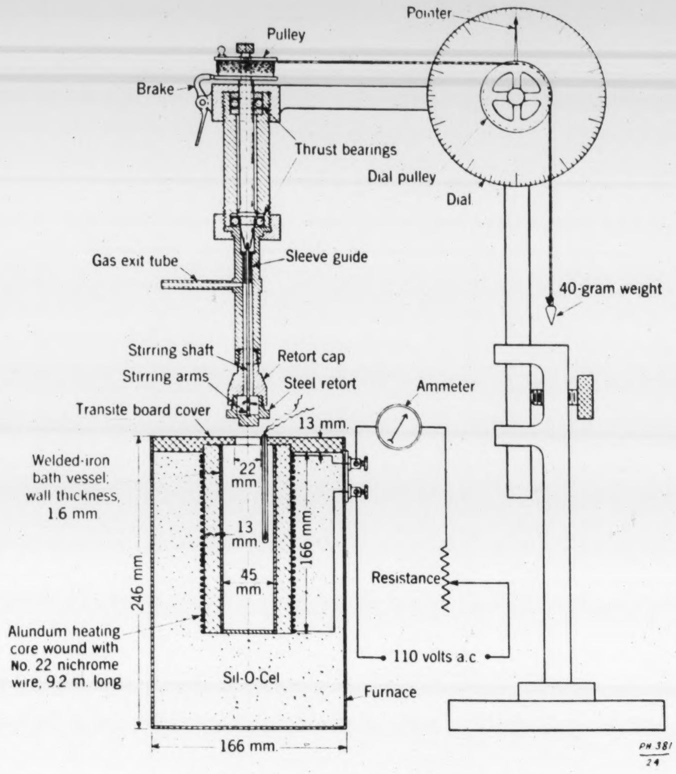

Pointer
Pulley
Brake
Thrust bearings
Dial pulley
Dial
Gas exit tube
Sleeve guide
40-gram weight
Stirring shaft
Retort cap
Stirring arms
Steel retort
Ammeter
Transite board cover
13 mm.
Welded-iron bath vessel; wall thickness, 1.6 mm
22 mm
13 mm
166 mm
45 mm
246 mm
Resistance
Alundum heating core wound with No. 22 nichrome wire, 9.2 m. long
110 volts a.c
Sil-O-Cel
Furnace
166 mm
PH 381 / 24

FIG. 1.—Gieseler Type Plastometer.

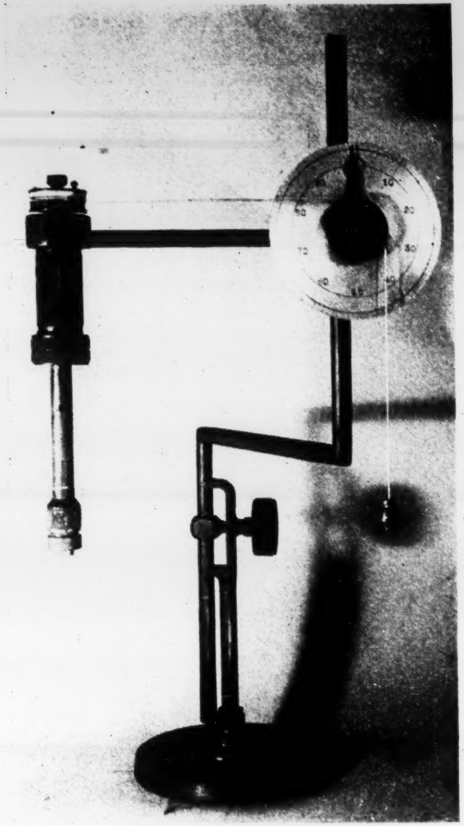


FIG. 2.—The Gieseler Plastometer.

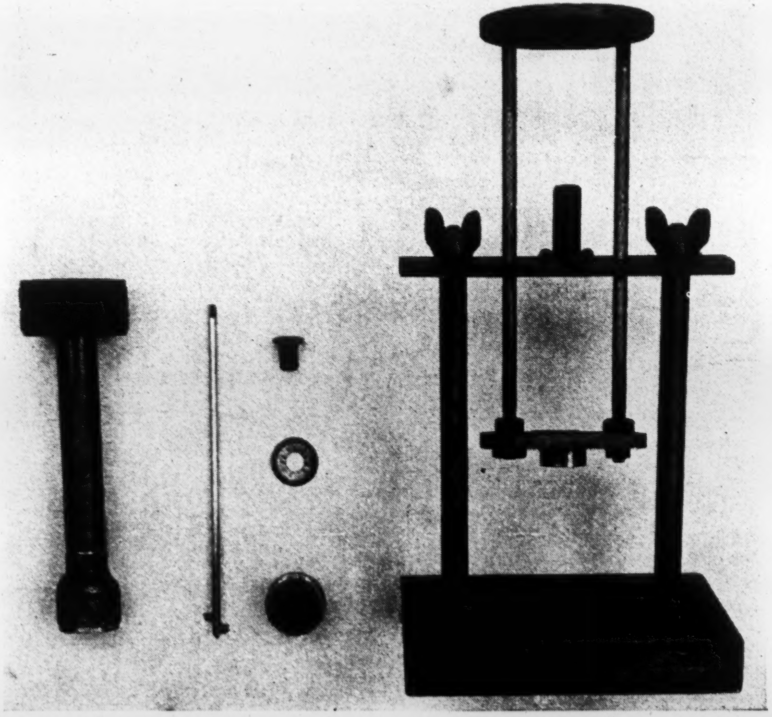
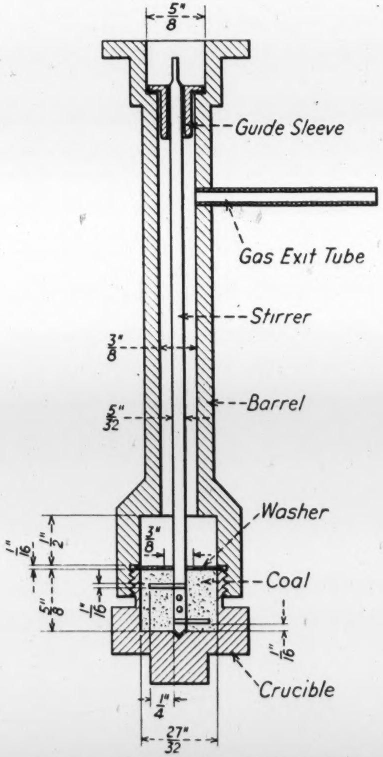


FIG. 3.—Parts of the Gieseler Plastometer Retort and the Loading Device.



5"/8
Guide Sleeve
Gas Exit Tube
Stirrer
3"/8
Barrel
5"/32
Washer
3"/8
Coal
1"/16
1"/2
5"/8
1"/16
1"/16
Crucible
1"/4
27"/32

FIG. 4.—Gieseler Retort Assembly.

depression which acts as a lower bearing for the stirrer. The stirrer is a piece of $\frac{5}{32}$-in. drill rod, possessing four rabble arms $\frac{1}{16}$ in. in diameter and $\frac{1}{4}$ in. long, placed at 90 deg. intervals around the stirrer shaft and $\frac{1}{8}$ in. apart, center to center. The lower arm is $\frac{1}{16}$ in. above the crucible bottom when the apparatus is assembled. The shaft of the stirrer moves in a retort barrel $\frac{3}{8}$ in. in inside diameter with the lower part bored out to form a chamber $\frac{27}{32}$ in. in diameter and $\frac{1}{2}$ in. high to allow for possible swelling of the sample. Between this chamber and the crucible is a steel washer with a central opening $\frac{3}{8}$ in. in diameter for admitting the stirrer shaft and allowing escape of the volatile products. The washer is held tightly between the crucible and the barrel when the retort is assembled. In the upper part of the barrel is a sleeve guide acting as a second bearing for the stirrer.

The plastometer head, shown in Fig. 2, consists of an axle mounted on ball-bearing races. The axle has a slot at the lower end to form a union with the stirrer shaft. A pulley is mounted on the upper end of the axle in such a manner that it can be disengaged from the axle to permit rewinding of the cord on the dial pulley without disturbing the stirrer and axle. The cord wound around this pulley on the plastometer head passes over a second pulley possessing a pointer. The pointer moves around a dial which is divided into 100 divisions for 360 angular degrees. A weight of 40 g. is suspended on the end of the cord and acts as a force to turn the stirrer. A brake attachment on the plastometer head prevents movement of the pulley except during readings.

The furnace used for heating the sample is an electrically heated unit containing "half-and-half" solder as a bath medium. The temperature of the bath is determined by a thermocouple inserted in the bath adjacent to the retort. By means of a variable resistance and a controller, not shown in Fig. 1, the rate of heating of the bath is arranged for 3 ± 0.1 C. per minute. The controller is a simplified form of the apparatus devised by Warren (13). This consists of a recording potentiometer modified so that a metal stylus, replacing the recording point, makes contact with either of two strips of aluminum foil on the recording chart roll. Contact with one strip operates a relay to decrease the resistance in the heating circuit and accordingly causes an increase in the heating rate. Contact with the other strip actuates the relay to increase the resistance and thus decreases the heating rate. The two strips of foil are fixed on the chart so that a heating rate of 3 C. per minute is followed.

Coal is packed in the crucible by means of the loading device shown in Fig. 3. A piston which just slides into the crucible and has an opening along the axis to permit the piston to slide along the stirrer is mounted in such a manner that a load of 10 kg. can be imposed to pack the sample. Coal is added and packed alternately until the top of the sample is level with the top of the crucible. The 10-kg. load is imposed for 15 min., a time which had been found to give a packing density that gave reproducible results in tests.

The coal sample used for testing is especially prepared for the Gieseler plastometer. It was found in some of the early work that the minus 60 mesh coal as ordinarily prepared for proximate analysis would not pack sufficiently to remain packed during assembly of the plastometer: minus 35 mesh coal was found to be satisfactory, however. In preparing the coal, after the gross sample has been crushed to about minus 20 mesh, a 50-g. portion is quartered out and the grinding to minus 35 mesh is done with a mortar and pestle.

PROCEDURE

After a sample has been packed in the crucible, the packing device is dissembled, the washer is placed on the crucible and the barrel is screwed on and tightened. The guide sleeve is inserted and then the retort is placed on the plastometer head, being sure that the axle in the head engages the stirrer.

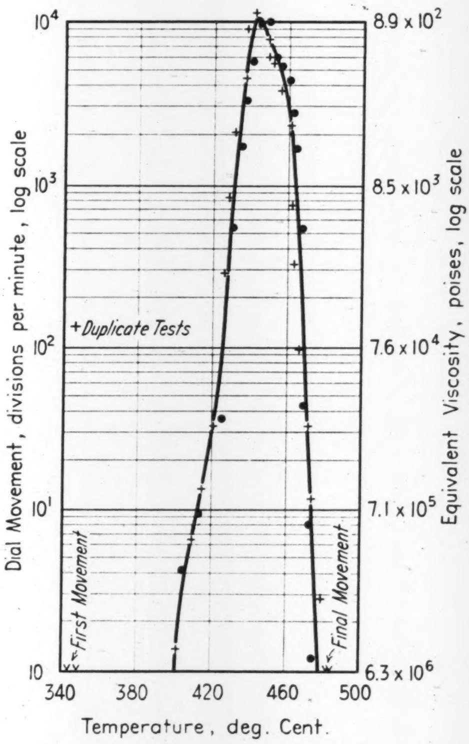


FIG. 5.—Duplicate Tests in the Gieseler Plastometer. Heating rate = 3 C. per min.

The furnace is previously heated to 300 C. The retort is immersed in the metal bath to a depth of 3 in. and the current going to the furnace is adjusted so that the retort and bath return to a temperature of 300 C. in 10 min. Thereafter the temperature rises at a rate of 3 deg. per minute and is regulated by the controller. The brake is released and the pointer of the dial pulley is set at 0. The temperature and time of the first detectable continuous movement of the pointer is recorded, the movement detectable being less than 0.1 division. After the first movement, readings are taken every 3 min. up to approximately 410 C., and every minute thereafter and recorded as divisions per minute after the movement exceeds 0.1 division per minute. If the coal does not show softening below 410 C., readings are taken every minute after the coal softens and in all tests readings are continued until the pointer again ceases to move.

RESULTS

When a test of a coal is completed, the data are plotted on semilogarithmic paper as shown in Fig. 5. The degree of fluidity attained by the coal, expressed as a rate of movement of the dial pointer, is thus shown in relation to temperature. Since the fluidity-temperature relationships vary for different coals, the rate of movement of the stirrer, expressed as divisions per minute, is meaningless unless tests of several different coals are available and the general properties of these coals are known. When tests of several different coals are available the results may be compared with one another and the relative plastic behavior of the coals can be expressed. In reporting results of Gieseler plastometer tests, characteristic points noted are: (*a*) the temperature of the first movement of the dial pointer, (*b*) the temperature at which the rate of dial movement becomes 5 divisions per minute, (*c*) the temperature at which the dial movement is greatest, along with the divisions per minute movement of the dial pointer at this temperature, (*d*) the temperature at which the pointer again ceases to move. It may be mentioned that the temperature at which the dial movement is 5 divisions per minute is noted because several investigators both in this country and in Europe have found an apparent relationship between this point in the plastic range and some other characteristics of coals present at this temperature.

Figure 5 also illustrates the reproducibility of results for this modified Gieseler type plastometer. The circles show the points obtained in one test and the crosses are the points found in a duplicate test while the curve drawn is the average curve for the two tests. Generally, duplicate tests show agreement within 5 C. for significant temperature points. The maximum rate of movement in divisions per minute which expresses the maximum fluidity may be duplicated within 20 per cent.

TABLE I.—PITCHES AND TARS USED IN THE CALIBRATION OF THE MODIFIED GIESELER TYPE PLASTOMETER.

Sample	Temperature, deg. Cent.	Viscosity, poises	Viscometer Used for Measurement[a]
A	25	5.90×10^7	Falling co-axial cylinder
	35	2.50×10^6	Falling co-axial cylinder
	45	1.92×10^5	Falling co-axial cylinder
	55	1.82×10^4	Large capillary rise
	65	2.24×10^3	Calculated from results at 55 and 70 C.
	70	8.41×10^2	Small capillary rise
B	70	6.67×10^2	Small capillary rise
C	25	4.34×10^4	Small capillary rise
D	40	3.2×10^3	Small capillary rise

[a] Falling co-axial cylinder method described at the 1941 Rheological Society meeting. Koppers capillary rise methods described in *Engineering News-Record*, November 21, 1935, and *The Laboratory* published by Fischer Scientific Co., Vol. VII, No. 5.

Calibration of the Plastometer and the Conversion of Results to Absolute Units:

Although the results of Gieseler plastometer tests, expressed in empirical units of divisions per minute dial movement, are expressions of the flow characteristics of coals and the temperature ranges during which these characteristics are present, these empirical units are quite meaningless by themselves for indicating the degree of fluidity attained. Thus, while it is certain that the coal in its fluid state is a heterogeneous system comprised of solid, liquid, and gas phases, it is desirable, nevertheless, to express the fluid state in terms of some absolute units. It was found possible to do this by calibrating the instrument, using several pitches whose viscosities had been measured in other instruments. Several samples were obtained and tested in the Gieseler plastometer at

TABLE II.—THE CONVERSION OF GIESELER UNITS, DIVISIONS PER MINUTE, TO ABSOLUTE UNITS, POISES.
Load, 40 g.

Gieseler Units, divisions per min.	Equivalent Viscosity, poises
0.1	6.4×10^7
1.0	6.3×10^6
10	7.1×10^5
100	7.6×10^4
1 000	8.5×10^3
10 000	8.9×10^2
15 000	6.6×10^2

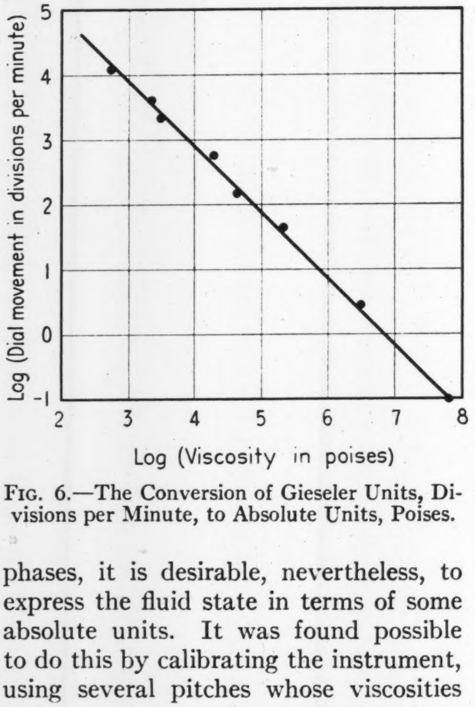


FIG. 6.—The Conversion of Gieseler Units, Divisions per Minute, to Absolute Units, Poises.

specified temperatures. The data for these samples are summarized in Table I.

The results of the calibration work are shown in Fig. 6, where the log-log relationship commonly used for viscosity curves is employed. Table II is a table of calibrations obtained by interpolation on the calibration curve of Fig. 6 and the logarithmic values converted to antilogarithms.

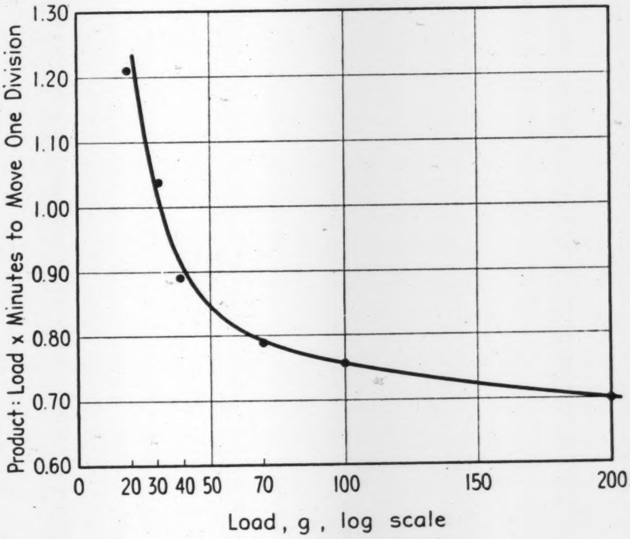


FIG. 7.—Change in the Rate of Movement of Stirrer with Change in Load Using a Calibrating Pitch.

The lowest movement of the Gieseler plastometer that can be measured is 0.1 division per minute and this value corresponds to a viscosity of 6.4×10^7 poises. The upper limit of measurement that can be obtained with any certainty is 15,000 divisions per minute, corresponding to 6.6×10^2 poises. It may be mentioned that the apparatus, when empty, shows a movement of the order of 35,000 divisions per minute. These results apply to operation of the plastometer using a load of 40 g. on the cord. To express the results in absolute units the term "equivalent viscosity" has been used since, as stated above, the fluid coal system is recognized as being heterogeneous.

The range of viscosities listed above for a calibration using a 40-g. load, 6.4×10^7 to 6.6×10^2 poises, may be increased by changing the load acting on the stirrer. Thus, loads greater than 40 g. can be used to study viscosities greater than 6.4×10^7 poises, and loads less than 40 g. can be used to determine viscosities less than 6.6×10^2 poises.

TABLE III.—CHANGE IN THE RATE OF MOVEMENT OF STIRRER WITH CHANGE IN LOAD USING A CALIBRATING PITCH.

Load, g.	Movement, divisions per min.	Product: load × minutes to move one division
20	16.6	1.21
30	28.9	1.04
40	45.0	0.89
70	85.0	0.82
100	131.0	0.76
200	285.0	0.70

Strictly speaking, for viscosities of true liquids, the rate of movement should be directly proportional to the load acting to cause movement. However, in the apparatus at the present stage of development, there are frictional resistances so that this relationship would be modified. Even if the frictional resistances were absent, it is improbable that coal would exhibit flow characteristics of true liquids. In order to test the possible use of loads other than 40 g. for measurements at the extremes of the range of viscosity noted above, several determinations were made using different loads from 20 to 200 g. and employing a pitch sample which had been found to show true viscosity characteristics. The results of these determinations are given in Table III and shown on Fig. 7. If there were no frictional resistance, the product of load and time to move a unit distance would be a constant. As expected, the results show that tests with loads less than 40 g. are greatly affected by mechanical resistance but at higher loads the effects are much smaller. Thus, if different loads are used, calibration of the plastometer for the different loads is necessary.

Typical Results of Tests:

Figure 8 presents results of tests on different ranks of coking coal. Generally, only low-volatile bituminous, medium-volatile bituminous, and high-volatile bituminous A and B coals show plastic properties during ordinary methods of carbonization. The results of tests on coals in these ranks show several generalizations in behavior. In regard to maximum fluidity shown during tests, the degree of maximum fluidity increases progressively as the rank of coal decreases, up to high-volatile bituminous A coals. Within the high-volatile A group a reversal of the behavior occurs and as the rank of coal decreases further, the maximum fluidity decreases again.

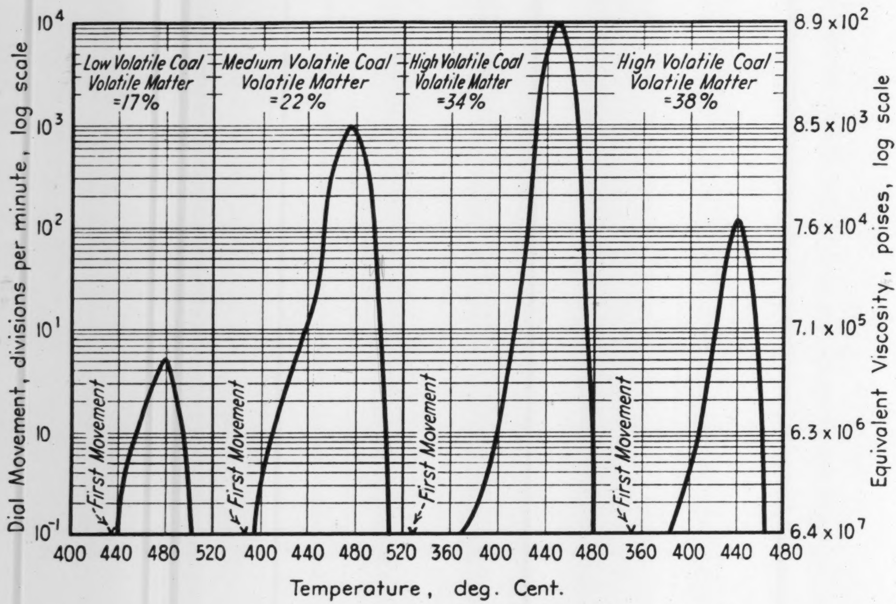
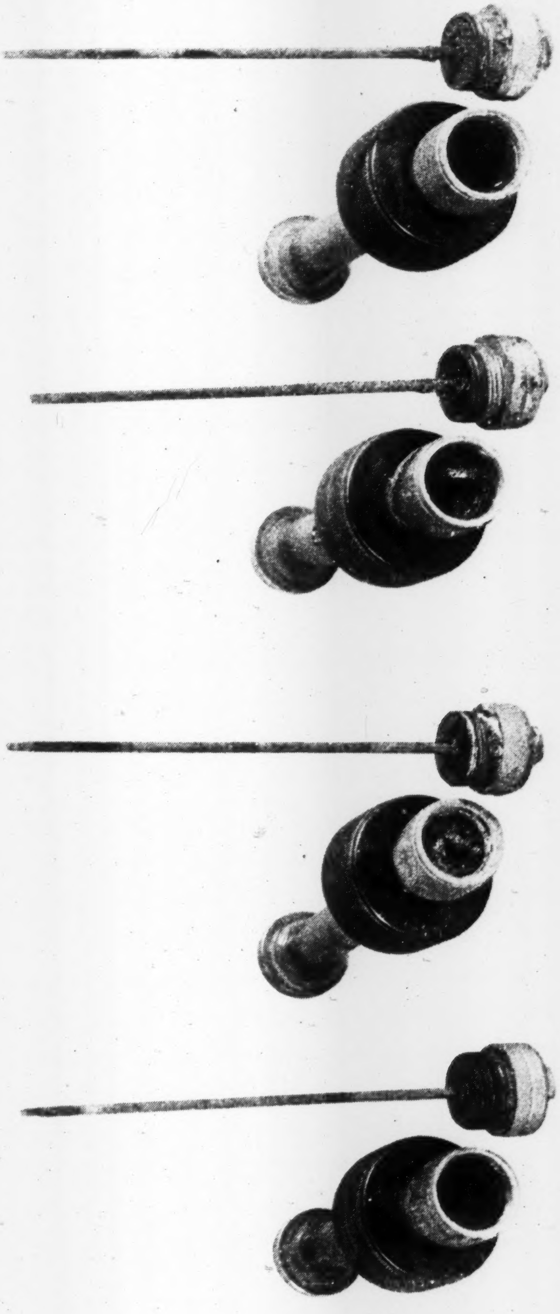


FIG. 8.—Gieseler Plastometer Tests of Different Ranks of Coking Coals, Heating rate = 3 C. per min.



Volatile Matter = 17 per cent

Volatile Matter = 22 per cent

Volatile Matter = 34 per cent

Volatile Matter = 38 per cent

FIG. 9.—Gieseler Plastometer Retort Residues of Different Ranks of Coking Coals.

Many coals in the high-volatile B group do not show any plastic characteristics in the Gieseler plastometer test. Another generalization is that the temperature range of plasticity varies in the same manner as the degree of maximum fluidity, increasing up to the high-volatile A group and then declining again within this group. The temperature interval may be 430 to 505 C. for low-volatile coals, 380 to 500 C. for medium-volatile coals, and 350 to 490 C. for high-volatile A coals, or temperature ranges of 75, 120, and 140 C., respectively. Within the high-volatile A group, when the degree of maximum fluidity and the temperature range of maximum fluidity begin to decrease, the temperature of resolidification declines more rapidly. Of course within any one rank, there is much variation. Some coals may show a beginning plasticity as low as 325 C. and others as high as 450 C. The end of the plastic interval may be as high as 515 C. or as low as 430 C. Generally, as the rank of coal decreases, the coal shows beginning plasticity at lower temperatures, the temperature of maximum fluidity decreases, and the resolidification temperature decreases, or, the temperatures of the entire plastic range are shifted to lower values.

The charts for these typical results show the movement in viscosity units as well as in divisions per minute. Low-volatile coals show a behavior equivalent to a viscosity of about 10^5 poises when the system is most fluid. Medium-volatile coals show a maximum fluidity equivalent to 10^4 poises. High-volatile coals may show a maximum fluidity equivalent to viscosities as low as 10^3 poises or less, while for still lower rank coals the equivalent viscosity at the most fluid stage increases again and may be 10^6 poises or more.

Figure 9 shows some representative retort residues which indicate the degree of swelling which may be found for various coals and shows how the swelling varies. It is noteworthy how similar the residues are in appearance from the low-volatile coal and the lower rank high-volatile coal. These residues also show why it is incorrect to think of the flow characteristics of plastic coal systems as being of the same character as those characteristics of pitches or tars. Further, the appearance of the residues indicates that some correction may be necessary in comparing the flow characteristics of different coals other than simple comparison of the measured movement of the stirrer in the plastic coal.

CONCLUSIONS

The modified Gieseler plastometer described in this paper has been used to test many different kinds of coking coals in regard to their plastic properties and has been found satisfactory for all the coals tested. The present form of the instrument has several changes in design over previous models which overcome deficiencies found for the use of the older types, particularly for measurements of the plastic properties of coals showing high fluidity. The procedure of operation has been found to permit excellent duplication of results for all coals tested.

The calibration work is an attempt to place the measurements of the plastic characteristics of coal on a scientific basis and permit results to be expressed in absolute units. The difficulty of expressing the results of tests in absolute units is acknowledged and the work is open to correction, or further modification. At least until such time when standard instruments are adopted for measurement of the plastic characteristics of coking coals, the expression of tests as presented here should serve to permit greater use of data now being obtained.

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DISCUSSION

MR. RALPH E. BREWER[1] (*presented in written form*[2]).—The authors are to be congratulated for their clear and concise treatment of the factors involved in measuring the plastic characteristics of coal and for their attempt to express these measured values in absolute units.

As the authors have stated, the plastic properties of coal must be measured during the time when the coal is actively decomposing. Such measurements, therefore, are of necessity conducted on "a heterogeneous system consisting of some solid infusible material, some fluid coal substance, and some gaseous matter present as bubbles in the fused coal." During these measurements, the proportions of solid, liquid, and gaseous materials present in this heterogeneous coal system change within the temperature interval in which the coal exhibits flow characteristics. Furthermore, the proportions of these three states of matter vary for coals of different rank and state of oxidation. For these reasons, no one of the usual terms relating to rheological properties of matter[3] accurately defines the coal system. However, the term "equivalent viscosity," suggested by the authors, appropriately represents the phenomena observed. This term should aid in bridging the gap between the data obtained by different methods for measuring the flow characteristics of coal and other natural and synthetic materials, and, at the same time, be acceptable to the rheologist until a better term is found to define the phenomena.

More details might be given in the test procedure, particularly with respect to the portion of time during the test when the brake is used. Alternately, this purpose could be served here if a reference were given to the Proposed Method of Test for Plastic Properties of Coal by the Gieseler Type Plastometer which has been published as information in the 1943 Report of Committee D-5 on Coal and Coke.[4]

A few errors, probably typographical, appear in the paper. The expression "uniformly heterogeneous" system, applied to tars, pitches, oils, etc., obviously should read "uniformly homogeneous." The coal used in the original Davis plastometer test procedure was "sized between 20 and 40 mesh," instead of "sized between 0 and 20 mesh," as stated in the paper.

I should like to ask some questions regarding the described test procedure. First, what is the order of duplicability in test results obtained with the plastometer in the calibration of the tars and pitches? Second, has any difficulty been experienced in binding of the stirrer during tests on certain coals with the use of the inserted iron washer? Our experience at the U. S. Bureau of Mines has been that some medium-volatile coals cause binding and sometimes a complete stopping of the rotation of the stirrer when the washer is used. Other medium-volatile coals with the washer gave higher maximum fluidity values than without the washer. Third, have enough data been obtained from tests made with and without the washer to show the relative maximum fluidity values for particular coals, especially for coals with high swelling properties?

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[1] Chemical Engineer, Coal Carbonization Section, Central Experiment Station, U. S. Bureau of Mines, Pittsburgh, Pa.

[2] Published by permission of the Director, Bureau of Mines, U. S. Department of the Interior, Washington, D. C.

[3] Standard Definitions of Terms Relating to Rheological Properties of Matter (E 24 – 42), 1942 Book of A.S.T.M. Standards, Part III, p. 857.

[4] See p. 301.

MR. B. P. MULCAHY[5] (*presented in written form*).—The writer agrees with the authors that the modified Gieseler apparatus offers the most promising method of studying plastic properties of coking coals. The authors point out that coal is such a transient material most methods employed in assaying its characteristics are necessarily empirical, and it is because of this fact that extreme caution must be used in interpreting all data.

In the paper presented, the authors have done an excellent piece of work in condensing and simplifying the description of the method and apparatus in determining plastic properties of coals. The apparatus they describe, however, differs not too greatly from that originally described by the U. S. Bureau of Mines to which reference has been made. The sample chamber is of larger volume than the original model and the washer above the coal charge has been added. As the authors mention, the enlargement of the sample space has improved the apparatus for the determination with high volatile, highly fluid coals.

The conversion of the units of measurement of plasticity from "dial divisions per minute" to the absolute units "poises," in addition to permitting a comparison with other materials of known plastic properties, also establishes the fundamental worth of the Gieseler apparatus for its work on coal. This is very important, for many of the methods of the coal chemist relate only to his own particular field, thereby narrowing the effect of proper interpretation. The effectiveness of the instrument for measuring coal plasticity, however, remains the same whether the results are reported in "poises" or in "dial divisions per minute."

A very important point which the paper does not develop very thoroughly is that each instrument will have its own frictional resistance characteristics. This is illustrated in Fig. 7 where we note that the point for the 40-g. load (the operational weight) is located at the critical point of the curve. In other words, it would seem advisable to have this point occur in the flatter portion of the curve.

The value of instruments, such as the Gieseler apparatus, lies in their general use so that considerable information can be obtained by different laboratories. In order to do this, however, the instruments must be so designed that they can be standardized to permit close checks between different men. The fundamental characteristic of this instrument is one of movement, and unless some limit is set for its permissible friction, different units will vary considerably in their measurements. It is proposed, therefore, that a maximum frictional resistance be established. In our own case we are able to get a sustained accelerating movement with a load of 4½ g. The upper speed limit is a function of still another factor.

In the "Conclusions" the authors state that the apparatus "has been found satisfactory for all coals tested." Also that it "permits excellent duplication of results for all coals tested." This might well be a point of issue, because highly fluid, high volatile coals offer an excellent example of the difficulties attendant with studying coal. A low or medium volatile coal will generally remain compact around the stirrer, so that its fluid properties can be fairly well measured. The highly plastic coal, on the other hand, froths and ascends to fill the crucible as well as the barrel, resulting in a false increase in speed of the stirrer due to a decrease in the density of the material surrounding the stirrer. This condition does not exist in the coke oven; when these exaggerated speeds are compared with the speeds of the low volatile coals we obtain a distorted picture, such as shown in Fig. 8. Because of the above factor the curves for the different coals do not represent actual relative fluidities.

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[5] Research Engineer, Citizens Gas and Coke Utility, Indianapolis, Ind.

We have had no particular difficulty in obtaining reproducibility of results on these high volatile coals, but at the same time, we feel that the results are very erroneous and must be so regarded. The instrument will have to be further modified to prevent these coals from expanding, and until such time we must not regard the fluidities of high and low volatile coals (fluid coals), as determined by this instrument as truly relative.

On page 1184, the authors mention that, generally, duplicate tests show agreement within 5 C. for significant temperature points. It is felt that this is too wide a variation to permit, particularly when comparing the "freezing points" of coals which all seem to freeze within a range of 35 C.

The authors mention, but do not discuss very thoroughly, the subject of packing of the sample. This factor deserves considerable attention because a great proportion of inaccuracies of the tests can be assigned to unreliable packing of the sample.

The use of the potentiometer to control the heating rate is a distinct improvement and renders the instrument more automatic in its operation.

In conclusion, the writer compliments the authors on their presentation, particularly on the establishment of the fundamental relationship with conventional fluid measurements. This is particularly heartening to coal chemists who have, too long, had to content themselves with make-shift methods in working with coal.

MR. GLENN C. SOTH[6] (*author's closure*).—I shall reply to Mr. Brewer's questions in the order in which they were raised, First, concerning the use of the term "uniformly heterogeneous;" "homogeneous" refers to a single phase system which does not include pitches and tars that contain dispersed free carbon in a liquid phase, so many tars and pitches are heterogeneous systems. However, the dispersion of the solid phase is such that any usual sample increment taken would show little if any difference in composition so that the term "uniformly heterogeneous" may be applied.

Concerning the order of duplicability, the results and the agreements in the calibration are actually much better than those obtained in actual tests on coal. I feel that the calibration is much more significant than any particular single test or pair of duplicate tests made on coal. I feel that the calibration is much more trustworthy than a test on coal itself, that is, the calibration using pitches and tars.

I should like to give a little information in answer to the question concerning the use of the inserted iron washer. The Gieseler method has been used in this country and in Europe by several investigators. They recognize the difficulty with a coal when it is heated at the ordinary rate as found in a coking oven—about 2 to 3 deg. per min.—and that a good many coking coals are going to swell and increase in volume. Many attempts have been made to restrict this swelling. All those attempts, as far as I know, have failed.

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[6] Assistant Research Chemist, Research Dept., Koppers Co., Kearny, N. J.

Washers have been used with small holes in them to make sieve plates. What happens is that the coal swells up into those holes, closing the holes, and a pressure is created inside the retort. We found that to be the case when we tried perforated washers. Instead of putting the perforations at scattered places throughout the plate, we thought we would put them at the center in one place. If the coal is going to swell, let it swell!

As far as binding with or without a washer in its present form, I cannot say, for this reason: We have used the washer in all our tests, feeling that it means a small restraint on the degree of swelling—and not a great restraint. So if the coal is swelling in a test like this, allow it to swell. So far with all the coal that we have tested since the adoption of this washer, we have not run into a case of binding.

I understand Mr. Brewer, of the U. S. Bureau of Mines, has run into instances, but with the coals we have tested—a considerable number—we have not had one case of binding.

I cannot answer Mr. Brewer's last question. Since we adopted the washer, we have not tested any coals without the use of the washer.

In answer to Mr. Mulcahy's discussion, the sample chamber of the Gieseler plastometer used by Koppers Co., Research Department is the same size as that used by the U. S. Bureau of Mines. The essential feature of the new design is a space above the sample chamber and the washer which allows highly fluid coals to swell without causing the stirrer to bind.

Frictional characteristics of instruments will vary as long as each laboratory builds its own instruments from whatever materials are on hand. Mr. Mulcahy's proposal that some maximum value for frictional resistance should be established is well taken. However, since frictional resistance in different instruments would still vary below the maximum, calibration of the instruments in terms of absolute units is the simplest way to check results from different instruments and laboratories. Thus, the effectiveness of results in terms of "poises" is an improvement over "divisions per minute." The use of a 40-g. load as the operating weight was found to be the most suitable for studying coals of different ranks. A heavier load, selected from the flatter portion of the calibration curve, would not be applicable for coals showing high fluidity. If other loads are used, calibration of the instrument to express the results in poises would permit comparison with tests under other loads.

The interpretation of results of tests is problematical. Different ranks of coal behave differently in regard to swelling during tests. Many investigators have considered the swelling during laboratory tests to be a fundamental characteristic of the coal just as much as the rheological properties. No way has yet been found to eliminate swelling during tests without obtaining high pressure development. For this reason we have believed it is better to refer to the "coal system" rather than to "fluid coal," and to admit differences in the system from time to time or for different coals due to swelling because some differences would be present whether or not swelling is prevented.

An analysis of duplicate tests has shown that for 83 coals, 94 per cent of all results agree within 5 C., 50 per cent of the results showed duplication of significant points within 2 C. The range of "freezing point" for these 83 coals was 75 C. compared to the 35 C. range cited by Mr. Mulcahy. We doubt whether this order of duplication can be improved materially in the present state of the art.

DEFORMATION UNDER LOAD OF RIGID PLASTICS

By Robert Burns[1]

Synopsis

Of importance in design considerations is the deformation under load of rigid materials such as hard rubber and plastics when used as insulating or structural materials in precision apparatus. This paper describes how the deformation apparatus of A.S.T.M. Tentative Method of Test for Distortion Under Heat of Molded Electrical Insulating Materials (D 648 – 41 T)[2] can be used to guide the design engineer. Data are given on the flow of several rigid materials under various combinations of pressure and temperature within the service range. The data also provide interesting information for the fundamentalist who may be concerned with yield points and simple shrinkage-flow relations, as well as the complex situation produced by the concurrent existence of shrinkage, inherent flow, and additional flow caused by the presence of water as a plasticizing agent.

Rigid organic materials possessing high resistance to deformation under load have been an essential part of precision electrical apparatus for many years. Of major importance are hard rubber and the phenolic thermosetting group including molded parts and laminates. Deformation under load, sometimes referred to as "cold flow," has always been a problem in hard rubber and in fact was an important incentive in the development of the phenolics since the latter, because of their thermosetting characteristic, retained substantial rigidity at high service temperatures.

In the early thirties, with the introduction of many of the so-called thermoplastics and the inherent possibilities of injection and extrusion processes, design engineers became interested in the merits of these newer materials, first with regard to the need for color in artistic designs and later for the more utilitarian uses. Since the start of the war, and the necessity for more flexibility in the substitution of materials, the thermoplastics have been given serious consideration for insulating and structural uses. For these purposes the designer must know how they will perform from a deformation standpoint.

Types of Deformation:

Change of dimension under pressure may be principally simple plastic deformation, as in hard rubber, polystyrene, methacrylate plastic, rigid vinyl compounds, or any nonhygroscopic material, or it may be a complex combination of shrinkage or swelling superimposed on plastic deformation as in the case of cellulosic derivatives. In these materials the situation is further complicated by the fact that contained moisture has a pronounced effect on the plastic deformation.

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[1] Bell Telephone Laboratories, Inc., New York, N. Y.

[2] 1942 Book of A.S.T.M. Standards, Part III, p. 1066.

The permanence and rigidity of most assembled apparatus involving plastics will depend upon the flow characteristics of the materials employed, how they are assembled and subsequently used. The data presented here will be principally directed to determining what are likely to be safe assembly practices, considering that most thermoplastics are high in deformation when compared to the thermosetting materials.

The device used for this study is the parallel-plate constant force device described in A.S.T.M. Tentative Method of Test for Deformation of Plastics Under Load at Elevated Temperatures (D 621 – 41 T).[3] The test specimen, conditioned if necessary, is placed between the parallel plates and the change in thickness observed at any desired combination of time, pressure, and temperature.

The parallel plate type of deformation test has been chosen for the following reasons: (*a*) it is far less difficult than the variable-force constant-deflection system which is implicit in most design applications; and (*b*) experience has shown that test results can be used with almost equal facility in predicting service performance. In his paper on relaxation, Hopkins[4] has described a variable-force constant-deflection method for determining relaxation of elastomers, but the problem is substantially more difficult with the so-called rigid materials, that is, materials possessing negligible elastic deformation.

As a practical design problem, a switching apparatus now using laminated thermosetting material of the phenolic type can be considered as an example. The apparatus consists essentially of metallic contacting members insulated from one another by thin sheets of the insulating material. Laboratory tests and field experience have shown that during the service life of the device, the individual insulators must not change in thickness more than 2 per cent due to deformation, shrinkage, or both, since greater changes will result in maladjustment of the contacts in service and consequent failure.

Let it be assumed that for engineering reasons, or perhaps due to wartime shortages, it is desired to replace the phenolic material with a thermoplastic. The latter materials are by definition inferior to the phenolics in resistance to deformation at high service temperatures, therefore, some compromises in other directions must be made to assure satisfactory performance.

So long as the thermosetting insulators were employed, exact knowledge of service temperatures was not required, but with the thermoplastics a more precise estimate must be made of the maximum temperature to which the apparatus will be subjected during its service life. Since the great majority of the thermoplastics are objectionably soft at temperatures in excess of 175 F., the differences between them at any reasonable upper indoor service temperature, for example, 120 F. or 140 F., while definitely sizable, are not sufficiently large to represent the difference between success and failure because the flow is too great in all cases when compared to thermosetting materials. Consequently it is a reasonable approach to choose whichever thermoplastic is most desirable from other standpoints, and to compensate for its relatively high flow by reducing the pressures used in assembly to a point where the deformation is of the same

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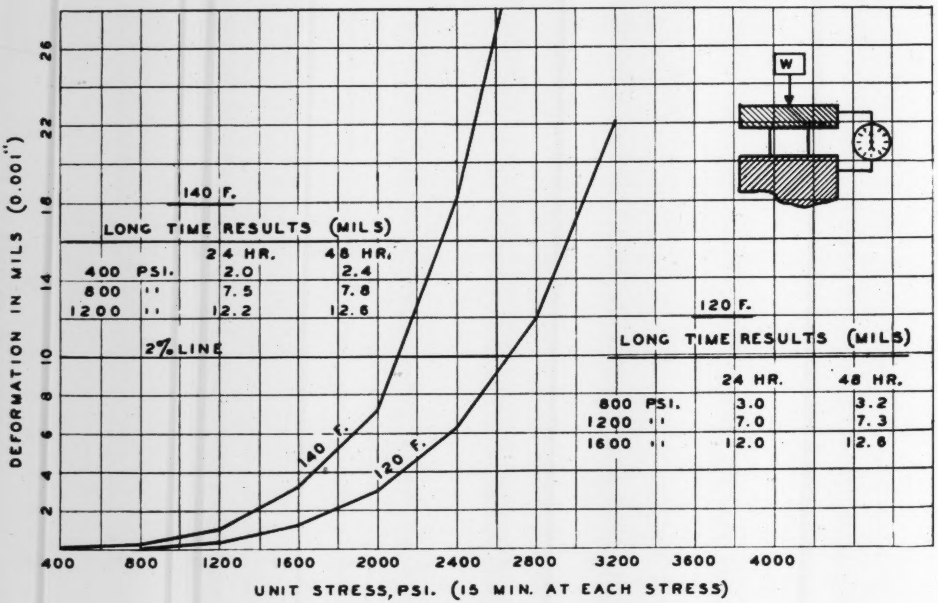
[3] 1942 Book of A.S.T.M. Standards, Part III, p. 1226.

[4] Irving L. Hopkins, "Relaxation of Rubber-Like Materials," see p. 1202.



140 F.

LONG TIME RESULTS (MILS)

	24 HR.	48 HR.
400 PSI.	2.0	2.4
800 "	7.5	7.8
1200 "	12.2	12.6

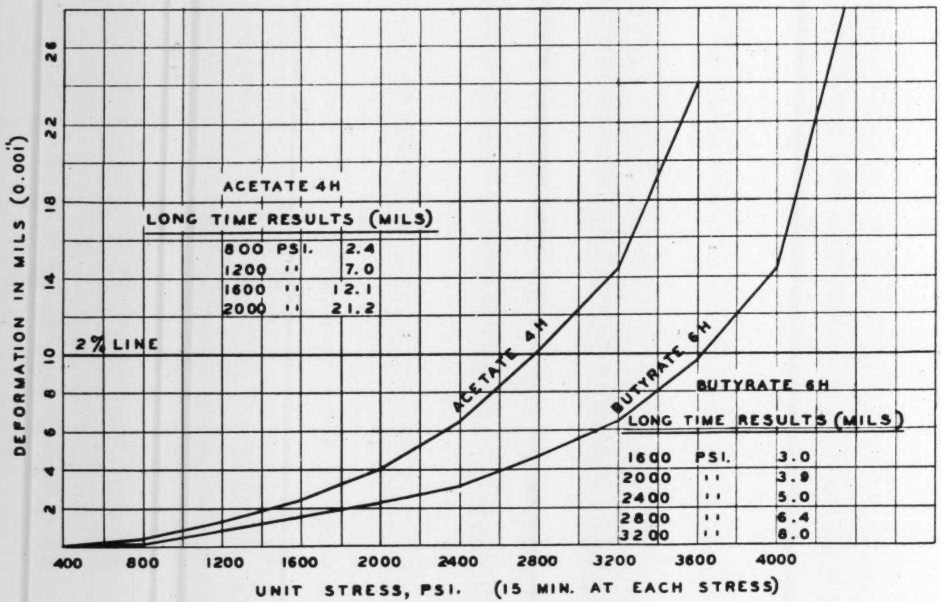
120 F.

LONG TIME RESULTS (MILS)

	24 HR.	48 HR.
800 PSI.	3.0	3.2
1200 "	7.0	7.3
1600 "	12.0	12.8

FIG. 1.—Cellulose Acetate Butyrate—H.

One half inch cube specimens no conditioning. Specimens exposed to test temperature for ½ hr. before applying first load.



ACETATE 4H

LONG TIME RESULTS (MILS)

800 PSI.	2.4
1200 "	7.0
1600 "	12.1
2000 "	21.2

BUTYRATE 6H

LONG TIME RESULTS (MILS)

1600 PSI.	3.0
2000 "	3.9
2400 "	5.0
2800 "	6.4
3200 "	8.0

FIG. 2.—Cellulose Acetate and Cellulose Acetate Butyrate (Hard Grades).

No conditioning. All results at 120 F. Long-time values are for 24 hr. only.

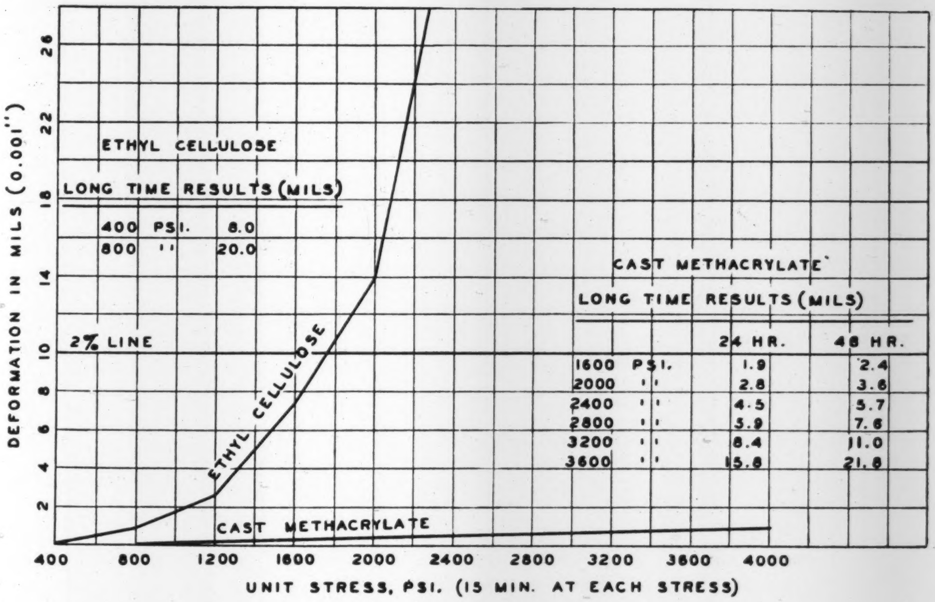


FIG. 3.—Ethyl Cellulose and Cast Methacrylate.
No conditioning. All results at 120 F.

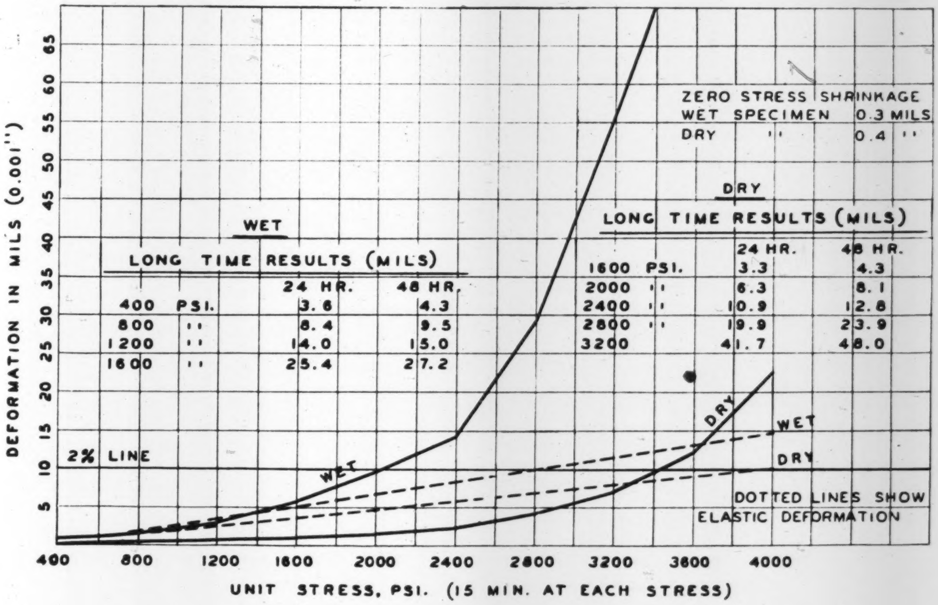


FIG. 4.—Cellulose Acetate—H 4.
"Wet" specimens were exposed to 90 per cent relative humidity at 85 F. for 48 hr. prior to testing.
"Dry" specimens were dried at 120 F. for 48 hr. period prior to testing. All results at 120 F.

order as that obtained with the thermosetting materials.

Assuming the substitute material has been chosen and the upper service temperatures decided upon, the apparatus illustrated in A.S.T.M. Tentative Method D 621 can be used to determine the maximum permissible pressure. Although it would be feasible to use the A.S.T.M. method making a 24-hr. test at each of many pressures, such procedure would be time-consuming and unnecessary. Satisfactory results can be obtained in a much shorter time by the following method: The first step consists of a relatively rapid exploration of the flow properties. This is done by placing a specimen in the machine at any desired temperature and, starting with a low pressure, say 400 psi., increase the load in suitable increments (for example, 400 psi.), holding for 15 min. at each increment, until the capacity of the machine (4000 psi.) is reached or until the 2 per cent limit is passed. The flow data (subtracting the elastic deformation) are plotted as ordinates and the stresses as abscissae. This curve gives a general picture of the deformation properties of the plastic at that temperature and also will disclose any sharp yield points as a function of pressure. A family of flow-pressure curves at various temperatures provides a complete short-time flow picture and will show the presence of a yield point, if any, as a function of temperature. With the exception of cast sheeting, test specimens used for this study were $\frac{1}{2}$-in. cubes cut from $\frac{1}{2}$ by $\frac{1}{2}$ by 5-in. test bars, compression molded.

Figure 1 illustrates the short-time flow properties of cellulose acetate of medium hardness, H. Elastic deformation is not included since in any practical application the elastic deformation is taken up immediately by the assembly pressure. It will be seen that this particular material exhibits no sharp yield at practical pressures and for temperatures of 120 and 140 F.

A line is now drawn at the ordinate representing 2 per cent deformation. It is obvious that all pressures beyond the intercept of this line with the flow curves can be omitted from further consideration. A group of pressures within the 2 per cent area are chosen arbitrarily for further exploration to determine long-time flow. These tests are occasionally continued for 48 hr. although experience has shown that a large portion of the flow takes place in less than 24 hr. Using the long-time flow data, the design engineer can be supplied with suitable assembly pressures.

Figure 2 gives short-time pressure-deformation curves and long-time deformation values for cellulose acetate of hardness 4H and cellulose acetate butyrate of hardness 6H. Figure 3 gives similar data on ethyl cellulose and cast methacrylate resin. Figure 4 illustrates flow of cellulose acetate of hardness 4H, under "wet" and "dry" conditions. The zero-pressure shrinkage on drying is also given. From these data a rough estimate of flow *per se*, flow enhanced by the plasticizing effect of water, and change of dimensions due to shrinkage can be made. In most cases the zero-pressure shrinkage is negligible. This type of analysis is of interest only in hygroscopic materials.

The choice of stresses for the long-time tests is suggested largely by experience with a given material. The data in Fig. 2 illustrate the futility of any broad generalizations. In the case of a material where no previous experience is at hand, a reasonable starting point is one half of the stress which produced the maximum permissible deformation in the short-time test. The data also emphasize the importance of time in any consideration of flow phenomena; hence the difficulty of applying rapid tests (such as ball indentation) in plastics design problems.

It is not the intention of the foregoing to present final engineering data on the materials tested nor to pass on their relative merits. The principal purpose here is to outline what is believed to be a useful procedure to obtain design information. The choice of temperatures, limiting values of permissible flow, and how the specimens should be conditioned, if at all, are all factors determined by the particular apparatus in mind, and where it is to be used. In addition, many thermoplastics are available in a wide variety of hardnesses, permitting the materials engineer substantial latitude.

DISCUSSION

Mr. M. B. Moore.[1]—It is my understanding that in this investigation, Mr. Burns used only the A.S.T.M. Standard Method D 621, which is a compression test on a cube specimen of the material with considerable lateral restraint at the loads. I should like to know whether any attempt was made to correlate these results with the results obtained using some other types of loading, particularly beam, shear, or tension loading.

In actual design problems, stresses other than simple compressive stresses will most certainly be encountered, and there is no assurance that the flow characteristics of a "rigid plastic" will be at all similar under different types of stress conditions.

Mr. W. N. Findley.[2]—Last year at this meeting we reported work done at the University of Illinois on creep tests of cellulose acetate in tension.[3] It is difficult to make an accurate comparison between our results and those reported by Mr. Burns because of the many differences in test conditions. However, the following observations on the two different tests of cellulose acetate may be of interest. At a stress of 2000 psi. in compression Mr. Burns' "dry" cellulose acetate (composition not reported) showed a deformation of about 1.6 per cent after 48 hr. under load at 120 F. Our tests at a stress of 2000 psi. in tension showed a deformation of about 4 per cent after 48 hr. under load at 77 F. and 50 per cent relative humidity. It is somewhat surprising that the higher temperature of Mr. Burns' test did not result in greater creep than in our test. However, this may be due to the lower moisture content or to differences in material or test method.

Our experience does not agree with Mr. Burns' experience that "a large portion of the flow takes place in less than 24 hr." The results of the tension creep tests referred to above indicate that tests continued for 6000 hr. at 1000 psi. stress may creep twice as far in 6000 hr. as in the first 48 hr. or a specimen at 2000 psi. stress may creep as much as 10 times as far in 6000 hr. as in 48 hr.

Mr. T. W. Dakin.[4]—I should like to ask Mr. Burns how much recovery he got from this deformation that he noted—what percentage of that deformation was recovered on release of the load.

Mr. Robert Burns[5] (*author's closure, by letter*).—With regard to Mr. Moore's question, this paper refers to specific applications in which parallel plate conditions are closely simulated. While it is certainly true that other types of designs might impose other than simple compressive stresses our experience has been that the parallel plate device gives us a fairly good picture of flow properties in general. However, there is no thought that this test possesses any particular sanctity. I believe any good flow test should suffice for obtaining design information.

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[1] Assistant Professor of Mechanical Engineering, Pratt Institute, Brooklyn, N. Y.

[2] Assistant Professor of Theoretical and Applied Mechanics, College of Engineering, University of Illinois, Urbana, Ill.

[3] William N. Findley, "Mechanical Tests of Cellulose Acetate—Part II on Creep," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 914 (1942).

[4] Research Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[5] Bell Telephone Laboratories, Inc., New York, N. Y.

In answer to Mr. Findley, most rigid plastics between parallel plates arrive at a point where the internal stresses are equal to those imposed by the test. At this point flow becomes negligible. This opposing force is certainly much less, and may be entirely absent, in the tension creep test. Consequently the lack of rigorous correlation is not surprising.

Referring to Mr. Dakin's question, this particular study did not include recovery data although we frequently include them in our flow work. The whole subject of recovery is very complicated, since the property depends on extent of deformation, temperature of deformation, temperature of recovery, time of recovery, and load if any. After we get the data, we do not know just what to do with them, especially in cases like those discussed in this paper where the plastics are not allowed to recover.

RELAXATION OF RUBBER-LIKE MATERIALS

By Irving L. Hopkins[1]

Synopsis

This paper is devoted to a study of the relaxation of rubber-like materials (elastomers); relaxation being defined as the decrease in stress in a material subjected to constant deformation. A method is described for measuring this property wherein each specimen is permanently fixed in its own test cell which may be subjected to any desired atmospheric conditions. The cells are inexpensive, making it practicable to test a large number of specimens over a long period. Test data on several materials are given.

The use of elastomers and non-rigid plastics as gaskets, where the material is held in a state of stress between substantially rigid metallic members, depends for its success on the ability of the material to support the stress for long periods. The evaluation of materials for this purpose should ideally involve the measurement of the stress in the gasket as a function of time; the relaxation test method described in this paper accomplishes this by measuring, without disturbing the specimen, the force exerted by it against the metallic retaining members at any desired time.

One of the methods heretofore used for evaluating gasket materials is the A.S.T.M. Tentative Methods of Test for Compression Set of Vulcanized Rubber (D 395 - 40 T)[2] in which the material is deflected under either constant load or constant deformation for a stipulated period, and the height of the specimen after its release is compared with its original height. The difference is a measure of the so-called compression set of the material. Another test which is useful but not directly applicable to the gasket problem is as specified in A.S.T.M. Tentative Methods of Testing Hard Rubber Products (D 530 - 39 T),[3] in which the gradual change in deformation is observed under constant load and temperature. Whereas this test uses the constant-load principle, the relaxation test herein described measures the change in force exerted by the specimen under constant deflection.

Figure 1 shows the test cell with the specimen in position between the top and bottom plates. The top plate is a loose fit in the inside of the cell wall, and is held against the three pins by the specimen; the bottom plate is drilled and pinned in place when the cell and specimen are assembled, as will be described later. The cell is therefore semipermanent, and can be conditioned and handled without any particular precautions.

To measure the force exerted by the

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[1] Bell Telephone Laboratories, Inc., New York, N. Y.

[2] 1942 Book of A.S.T.M. Standards, Part III, p. 1339.

[3] *Ibid.*, p. 1389.

specimen at any time, the cell is put in a compression testing machine and a load-deflection curve is run on the top plate of the cell relative to the plane of the bottom of the cell. This curve divides into two parts. The first involves the compression of the whole cell, including the elasticity of the pins and the compressibility of the cell walls. As soon as the load is off the pins, the load-deflection curve has the characteristics of the specimen rather than of the cell, and by extrapolating the curve back to the axis of ordinates we find the point of intersection giving the load at which the material under test is in equilibrium. The success of this method depends upon there being a considerable difference between the compressibility of the cell and that of the specimen. A typical load-deflection curve is shown in Fig. 2. The total compression of the cell and specimen during the measurement is in this case less than 3 mils, and it is assumed that this will not, during the few minutes it takes to make the measurement, affect the final relaxation results appreciably.

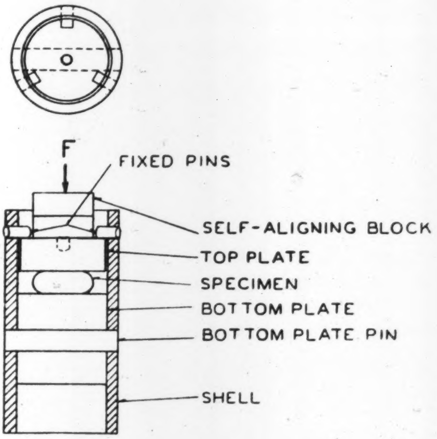

FIG. 1.—Relaxation Test Cell.

Preparatory to a test, each cell is assembled without any specimen, the bottom plate is pushed up against the top plate, and the available space in the bottom of the cell is measured with a depth gage, as shown in Fig. 3. The space to be occupied by the compressed

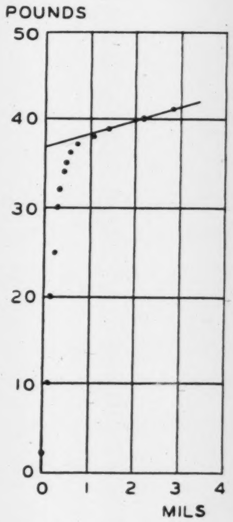

FIG. 2.—Typical Load-Deflection Curve of Relaxation Test Cell.

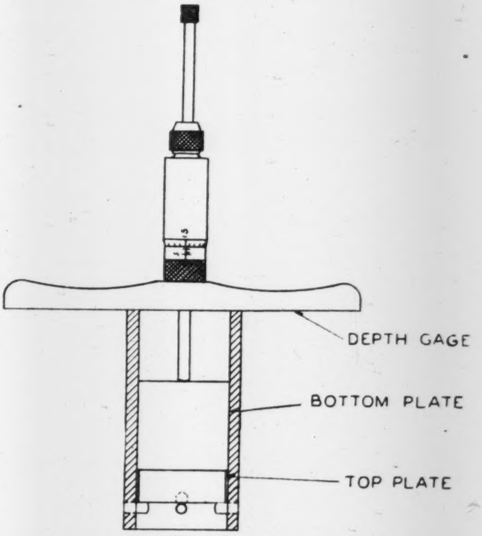

FIG. 3.—Measuring Available Space in Relaxation Test Cell.

specimen is calculated, based on the desired constant deflection, and the result subtracted from the available space; the difference will then be the open space in the bottom of the cell, and this amount is made up by means of a series of shims. To assemble the cell, the specimen is then centered upon the bottom plate, and the top plate centered on the specimen. The outer shell is carefully lowered over these parts and the lower plate pushed up until the top plate is against the pins. The cell is inverted and the shims put on top of the bottom plate. The assembly is then compressed in a drill vise, a $\frac{1}{4}$-in. hole is drilled through the cell wall and the bottom plate, and a pin is pushed in as shown in Fig. 4. The cell is then removed from the vise and the force exerted by the specimen is measured immediately. The cell then can be stored at any desired conditions for subsequent measurement.

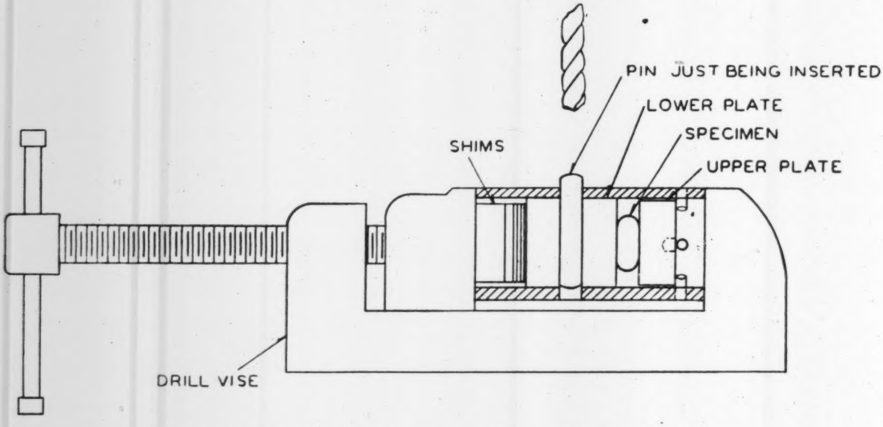
PIN JUST BEING INSERTED
LOWER PLATE
SPECIMEN
SHIMS
UPPER PLATE
DRILL VISE

FIG. 4.—Method of Assembling Relaxation Test Cell.

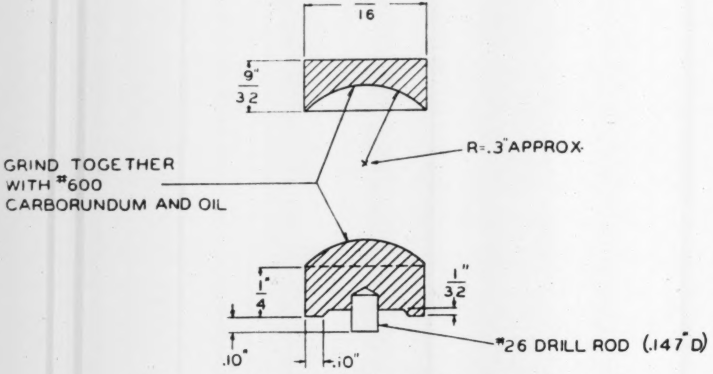
11"
16
9"
32
R=.3" APPROX.
GRIND TOGETHER
WITH #600
CARBORUNDUM AND OIL
1"
4
1"
32
.10"
.10"
#26 DRILL ROD (.147" D)

FIG. 5.—Self-Aligning Plate for Relaxation Test.

The technique of measuring the load is the most critical part of the procedure. The machine which has been used is a Meyers analysis hardness testing machine in which load can be added by simply running the weight out on the beam. A dial gage graduated to 0.001 in. is used to measure the deflection. The cell is put in the machine and the self-aligning member adjusted. The self-aligning member is shown in Fig. 5; this is an important detail, as it should be free enough to align itself at the beginning of the test and then have enough friction to prevent slipping even if the load is somewhat eccentric. If it slips, an unsatisfactory curve such as shown in Fig. 6 is obtained.

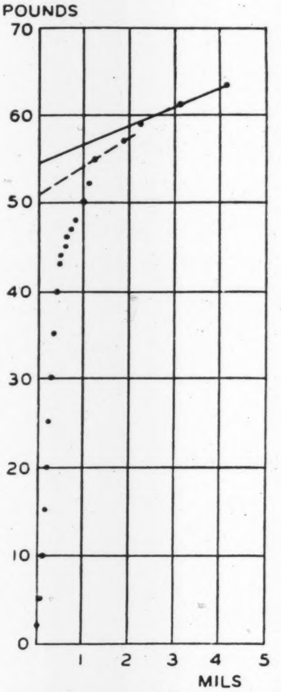

FIG. 6.—Load-Deflection Curve of Relaxation Test Cell with Improper Alignment of Self-Aligning Block.

A pre-load of about 4 lb. is applied, the base of the machine is tapped with a rawhide hammer in order to eliminate the effect of friction, and the dial gage is set to zero. Increments of load are then applied, after each of which the machine is tapped with the hammer, and the dial reading is plotted against the load. It is of considerable assistance if one knows approximately the load sustained by the specimen, but at any rate when the break appears in the curve enough more points are taken to establish accurately the slope of the second part of the curve, and also to be sure that the curve will not be of the kind shown in Fig. 6. Imperfect curves may also result from the upper plate being in contact with one of the side walls, or from dirt between the machine and self-aligning blocks, on the top plate, or underneath the cell.

Figure 7 shows some of the data obtained with cells of this kind. The materials were rubber and synthetic rubbers, including polysulfide. The specimens were pileups, with a nominal total height of $\frac{1}{2}$ in., of $\frac{1}{2}$-in. diameter disks, and were compressed 50 per cent. Several things of general interest are noticeable in Fig. 7. In the first place, noting that the solid lines represent tests made at room temperatures and the dotted lines tests made on specimens stored at 120 F. between tests, the increased rate of relaxation as the temperature is increased is well demonstrated. The particular compounds of rubber, Buna S, Buna N, butyl rubber, and chloroprene (1) are all fairly good. The polysulfide relaxes more rapidly at both temperatures. It also should be noted that if one had attempted to compare these materials after only a day or so, one might have concluded that the polysulfide is much better than it really is. Figure 8 shows the same rubber and polysulfide curves, with various non-rigid plastics. Here it is shown that thermosetting polyvinyl butyral is better than polysulfide. Ethyl cellulose did not stand up well, being only slightly superior to polysulfide. Polyisobutylene relaxed very quickly, even at room temperatures. Figure 9 shows the same

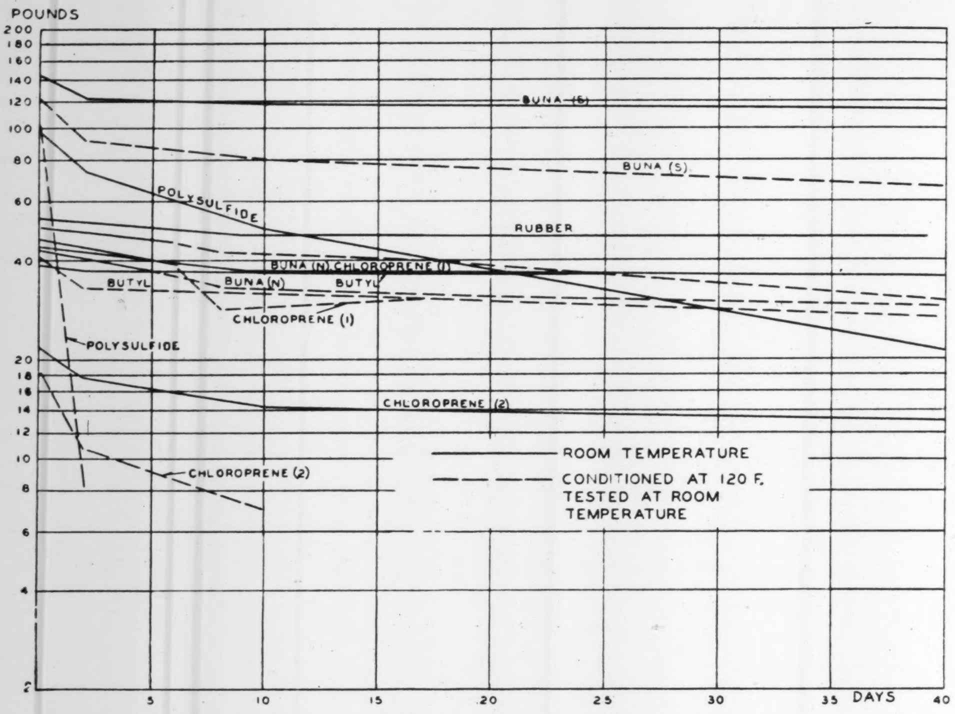

FIG. 7.—Relaxation of Rubber, Allied Materials, and Polysulfide.
Pounds force exerted by ½ in. pile up of ½-in. diameter disks compressed 50 per cent.

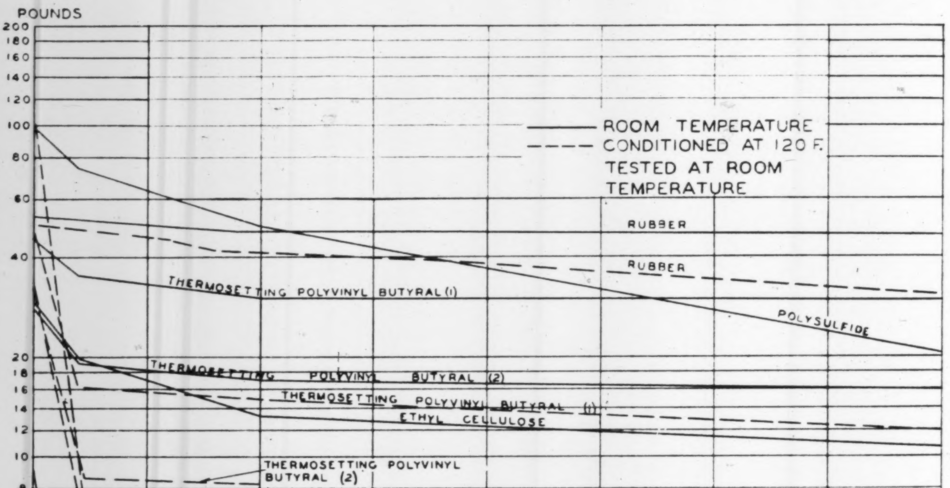

FIG. 8.—Relaxation of Rubber and Non-Allied Nonrigid Plastics.
Pounds force exerted by ½ in. pile up of ½-in. diameter disks compressed 50 per cent.

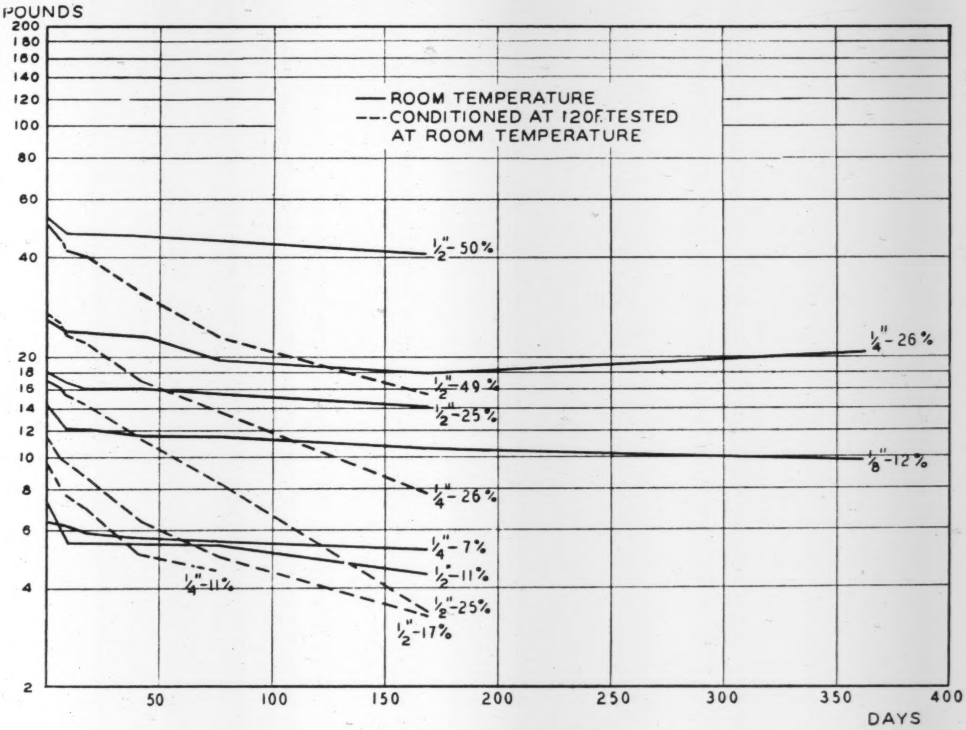

FIG. 9.—Relaxation of Rubber.
Pounds force exerted by pile ups of various heights at various compressions.

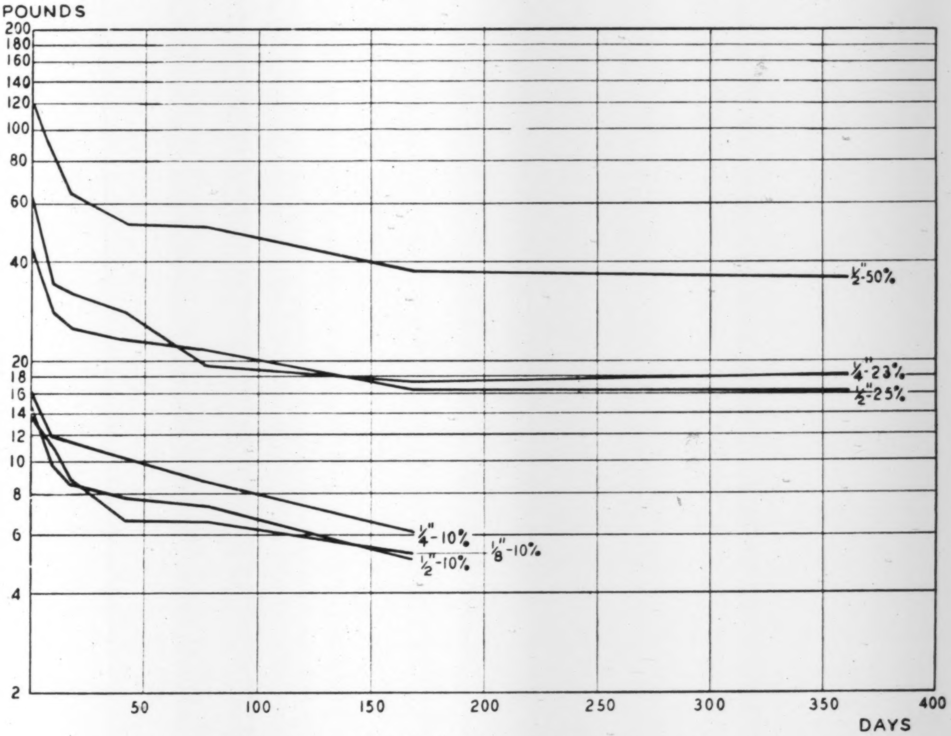

FIG. 10.—Relaxation of Polyvinyl Chloracetate at Room Temperature.
Pounds force exerted by pile ups of various heights at various compressions.

natural rubber as before, in various heights of pileup, compressed to different amounts. Here it will be seen that all the room temperature curves are roughly parallel, and that all the 120 F. curves are also parallel. Since the plots are on semi-log paper, this indicates that the percentage decrease in stress in a given material in a given time is a function only of the temperature, which simplifies considerably the evaluation of a material. Of course this remains to be demonstrated for each material, but Fig. 10 shows it to be true for polyvinyl chloracetate at room temperatures and similar data on Buna N and chloroprene show it to be approximately true for these materials also.

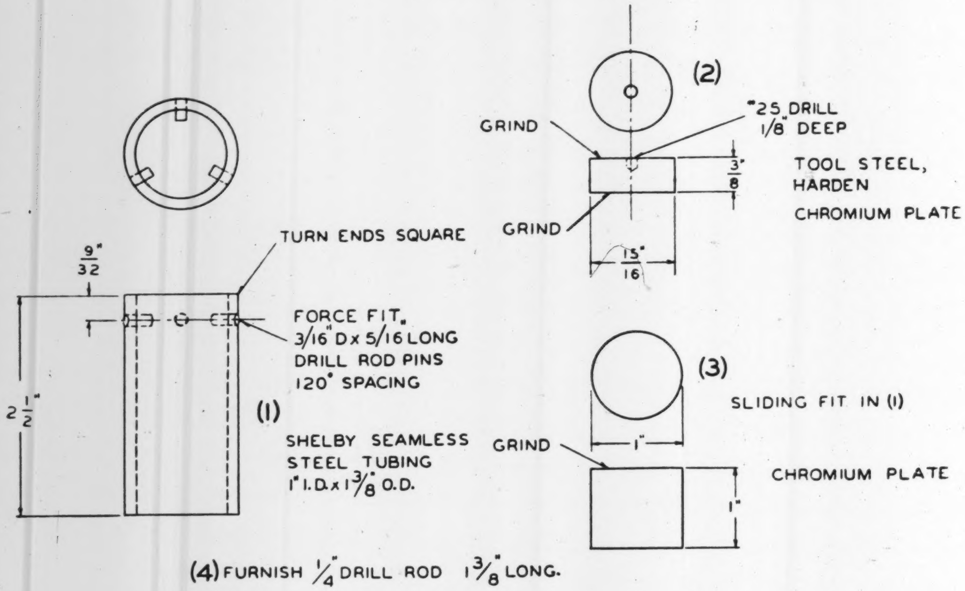
(2)
GRIND
#25 DRILL
1/8" DEEP
3"/8
TOOL STEEL,
HARDEN
CHROMIUM PLATE
GRIND
15"/16
TURN ENDS SQUARE
9"/32
FORCE FIT.
3/16" D x 5/16 LONG
DRILL ROD PINS
120° SPACING
2 1/2"
(1)
SHELBY SEAMLESS
STEEL TUBING
1" I.D. x 1 3/8" O.D.
(3)
SLIDING FIT IN (1)
GRIND
1"
CHROMIUM PLATE
1"
(4) FURNISH 1/4" DRILL ROD 1 3/8" LONG.

FIG. 11.—Details of Improved Relaxation Test Cell.

It should be borne in mind that the compounds tested for this paper were used to present the test method, and should not be construed to pass on the relative merits of the different base materials. The design engineer will of course have a wide variety of compounds of each material to choose from. Future work may show a correlation between the short-time results of this test and the A.S.T.M. tests mentioned earlier. It is not suggested that this method of test will be applicable to specifications work, but it has considerable possibility as a laboratory tool.

A working drawing of the test cell is given in Fig. 11.

DISCUSSION

Mr. D. P. Meigs.[1]—May I inquire what would be the relative field of application of this test as compared with the conventional test for deformation under load?

Mr. I. L. Hopkins.[2]—I think the two tests might correlate. I should regard the relaxation test as a research tool, both because it gives a quantitative measure of the actual falling off in pressure in a gasket, and because it is essentially a long time test as now arranged.

Mr. Meigs.—By that do you mean that this would be a test which might possibly be substituted for the cold-flow test at perhaps less cost to the investigator in order to obtain the same data?

Mr. Hopkins.—No; the relaxation test is more difficult and time consuming to set up, and is hardly suited to be used as an inspection test.

Mr. Meigs.—I could not help but get the impression from your discussion of the manner of obtaining data on this that it does require quite a good bit of attention on the part of the worker.

Mr. J. H. Ingmanson.[3]—I assume that Mr. Hopkins' paper is primarily a description of equipment, but nevertheless I am prompted to ask whether his results indicated an inferiority for, shall we say, buna-S compounds as compared with rubber for such applications as gasket materials.

Mr. Hopkins.—That is indicated in the curves that I showed but those are the only rubber and Buna-S compounds I have tested. It is probable that by changing compounds the order of the curves could be changed.

Mr. A. W. Carpenter.[4]—I think that question is very pertinent. For identification purposes Mr. Hopkins has apparently labeled his curves according to the base material in the particular sample that he had. This does not mean that the properties of that base material could not be substantially modified. Although the curve given may be labeled buna-S, it is quite possible that one would test materials of which he did not know the precise composition. He might merely know that it was a compound which was made of a certain type of material but, as we all know, those materials are subject to individual variation by the use of admixtures and different methods of processing, and so forth. Consequently, the curves may not necessarily be typical of that material, but merely for identification purposes are called by the name of the base material.

Mr. Ingmanson.—I think we should assume that Mr. Hopkins' data are purely qualitative and only illustrate the development of a test method, is that correct, Mr. Hopkins—primarily, shall we say?

Mr. Hopkins.—The data should not be taken to show anything about the base materials as classes.

Mr. M. L. Macht.[5]—I should like to

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[1] Chemical Engineer, Superpressure Division, American Instrument Co., Silver Spring, Md.

[2] Bell Telephone Laboratories, Inc., New York, N. Y.

[3] Vice-President, The Whitney Blake Co., New Haven, Conn.

[4] Manager, Testing Laboratories, The B. F. Goodrich Co. Akron, Ohio.

[5] Chemical Division, Plastics Dept., E. I. du Pont de Nemours and Co., Inc., Arlington, N. J.

know whether this work included orientation experiments which would give any idea of the effect of the previous history of the sample, particularly with regard to strain that might have existed.

MR. HOPKINS.—We have done no work on orientation nor on previous stress history. The samples were all cut from sheets about $\frac{1}{8}$-in. thick, and the specimens were generally pileups of disks cut from these sheets. The stress during test was therefore parallel to the direction of molding or calendaring pressure.

MR. F. E. HOFFMANNS.[6]—Mr. Hopkins mentioned in his paper the fact that a change in the room temperature may have affected the curves. Is it assumed that all the different types of samples changed the same amount for the temperature change or are the curves used for comparative purposes only?

Would the curve for each material change the same amount if the room temperature were kept constant? With one material the line would straighten out more than the other, would it not?

MR. HOPKINS.—The materials were generally different in sensitivity to temperature, and the curves show that there is certainly a difference between materials in the degree of relaxation.

MR. CARPENTER.—We have wondered for a number of years with respect to rubber whether it would not be better to do just what Mr. Hopkins has done—to measure the decay of load rather than to apply a constant load, as most of our tests do, and measure the deflection or flow, that is, the compression set. This apparatus appears to provide a good method for doing that. I tried one time to design a fixture in which relaxation was measured and I found that I was really up against something both from the point of view of mechanical arrangement and cost. Mr. Hopkins has certainly found a simple, convenient way of doing a very interesting piece of work.

MR. W. N. FINDLEY.[7]—Figures 9 and 10 of the paper show the progress of relaxation for different amounts of initial compression. I wonder whether any correlation was found between the rate of relaxation and the initial compression, such as the relation between stress and rate of creep in the case of creep tests.[8] If such a relationship, either graphical or analytical, can be demonstrated it would make a valuable addition to the paper. It would indicate that the rate of relaxation could be predicted for other values of initial compression than those tested.

MR. HOPKINS.—It appears that the rate of relaxation can be predicted, at least for some materials. The parallelism of the curves at a given temperature in Figs. 9 and 10 is independent of both the height-diameter ratio of the unstrained pileup and the strain. If this proves to be generally true the ratio of relaxation to initial stress will, for a given material, be some particular function of time and temperature which will need to be determined only once for each compound.

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[6] Research Engineer, The Patterson Foundry and Machine Co., East Liverpool, Ohio.

[7] Assistant Professor of Theoretical and Applied Mechanics, College of Engineering, University of Illinois, Urbana, Ill.

[8] W. N. Findley, "Mechanical Tests of Cellulose Acetate-Part II on Creep," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 914 (1942).

IMPACT TESTING OF PLASTICS—I. ENERGY CONSIDERATIONS

By David Telfair[1] and Howard K. Nason[1]

Synopsis

Impact strength values obtained by the standard Izod and Charpy test methods do not reliably indicate the behavior of a plastic part when subjected to impact stresses in service nor do they accurately rank different plastic materials with respect to their ability to resist fracture when exposed to such stresses. An analysis of the energy relationships in these tests accounts for some of the discrepancies, and a modified test, which measures the energy required to just initiate fracture in a test specimen, is proposed. The results obtained by this modified test rank materials in the order of their known shock-resisting qualities, and correlate well with service experience.

The shock-resisting properties of organic plastics have been evaluated on low-capacity pendulum instruments of the Charpy and Izod types for at least 20 yr. The present A.S.T.M. Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 - 41 T)[2] was originally intended for comparing electrical insulating materials of generally similar composition and physical characteristics. The test has been of great value for this purpose.

However, during the past decade, diversified expansion of the industry has made available a greatly increased variety of plastic materials. For a number of reasons, including simplicity and cheapness of apparatus, rapidity with which data may be obtained, apparent ease of operation, and apparent reliability of results, the standard impact test has been widely adopted for control, inspection, specification and research testing of these materials.

The pendulum type of test possesses two fundamental disadvantages:

1. Test values must be obtained on a standard specimen and can be compared only on the basis of that standard specimen; they cannot be referred to other sections, have no absolute physical significance, and cannot be utilized in design calculations. This fact has been realized from the inception, many years ago, of work on impact testing in the Society's Committee D-9 on Electrical Insulating Materials (**1, 2, 3, 16**).[3]

2. Impact test values do not measure the true energy required to fracture the specimen nor do they reliably indicate relative shock resistance of materials which differ markedly in composition or mechanical properties. This, also, has been pointed out repeatedly (**4**).

Suggested solutions to the first objection have recently been published by Callender (5), who proposes a method for obtaining impact values which are valid for specimens of any section, and by Koon (6), who proposes a method for calculating a "modulus of impact" from A.S.T.M. impact test (D 256) results.

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[1] Research Physicist, and Assistant Director of Research, respectively, Plastics Division, Monsanto Chemical Co., Springfield, Mass.

[2] 1942 Book of A.S.T.M. Standards, Part III, p. 1071.

[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1219.

It is the purpose of this paper to describe the results of research into the second disadvantage and to propose a modified impact test which will afford a more reliable measure of the shock-resistance of present-day plastic materials.

NOTE.—Callender, whose researches were published after most of our experimental work was completed, accomplishes a similar result by the application of similar physical reasoning, and we are glad to acknowledge his contribution to the field. Both of us, however, were anticipated by Church and Daynes (7), whose work on Ebonite came to our attention only recently and was also overlooked by Callender until after his work was completed. Although, Church and Daynes used a different experimental technique, their method of analysis is remarkably like ours, and is shown by our results to apply beautifully to a wide range of plastic materials. We acknowledge, without question, their priority of thought in this field.

PHYSICAL CONSIDERATIONS

An analysis of the A.S.T.M. impact test (D 256) shows that the value reported as "energy-to-break" the specimen is really the sum of the energies consumed by several mechanisms. The more important of these are:

1. Energy to initiate fracture of the specimen.
2. Energy to propagate the fracture across the specimen.
3. Energy to deform the specimen plastically.
4. Energy to throw the broken ends of the test specimen.
5. Energy lost through vibration of the apparatus and its base, and through friction.

In a brittle material, the tearing energy will be very small relative to the fracture energy, whereas in a tough, ductile, or fiber-filled material tearing energy may be very large relative to fracture energy. The notch, which is always present in the standard test specimen, serves to concentrate the stress and largely prevents plastic deformation; hence, energy consumed in plastic deformation is insignificant in most cases. The energy absorbed by the thrown ends of the broken specimen may represent a large fraction of the total energy used in the test (5, 8, 9), especially in the case of brittle materials. Energy losses due to vibration of the apparatus may be large in testing metals (10), but are apparently negligible for the standard plastics test (1). Friction losses are largely eliminated by careful design and operation of the apparatus (11).

The investigation herein reported was undertaken to evaluate the magnitude of the above energies and to clear up some of the inconsistencies observed when testing plastics of different composition by the standard impact test. For example, a phenolic composition containing mica or asbestos filler has a greater impact strength, as determined by the A.S.T.M. test, than does one containing wood flour, although the former is known from its behavior in fabrication and service to be considerably less shock resistant.

Just as the usefulness of a part in service is essentially ended by a static load which stresses it beyond its yield value, so is its capacity for further service destroyed by a shock load of sufficient magnitude to produce cracking. Hence, it seemed reasonable to consider cracking as a criterion of failure, and to determine the impact energy required to just crack a specimen, as previously proposed by Mains (12). This was done by a modification of the standard Izod test, in which the initial elevation of the hammer

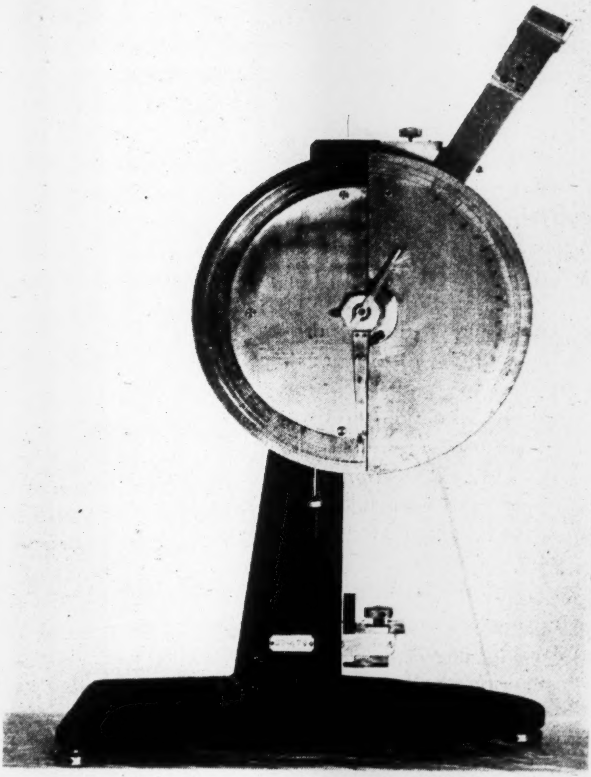

FIG. 1.—Standard Impact Testing Machine.

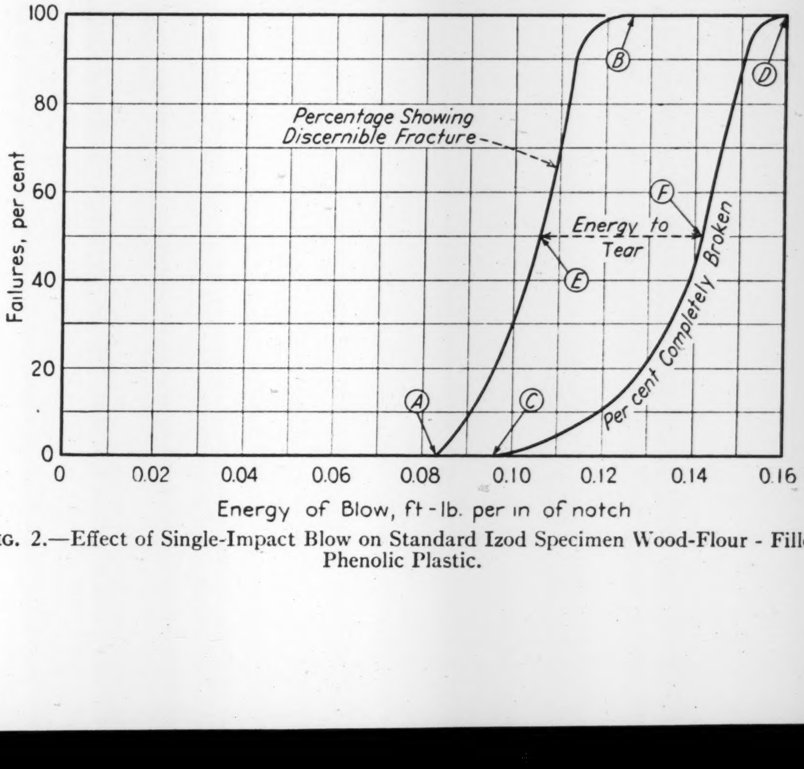

FIG. 2.—Effect of Single-Impact Blow on Standard Izod Specimen Wood-Flour - Filled Phenolic Plastic.

was varied so as to vary the kinetic energy available at the moment of impact.

NEW TEST METHODS

The standard impact apparatus was provided with an auxiliary dial which indicates the potential energy of the pendulum at any desired initial elevation (Fig. 1). Thus, the reaction of the specimen to blows of any desired kinetic energy could be studied. Best results were obtained by a statistical energy-to-fracture method, in which several groups of specimens were subjected to single-blow impacts of predetermined energy, and the fraction of the specimens in each group showing (*a*) a barely discernible fracture, and (*b*) complete break, was plotted as a function of the energy of the blow. An example is shown in Fig. 2. From these curves the following information may be obtained:

1. Shocks of less than 0.083 ft-lb. per in. of notch will crack none of the specimens (*A* in Fig. 2).
2. Shocks of greater than 0.125 ft-lb. per in. of notch will crack all of the specimens (*B* in Fig. 2).
3. Shocks of less than 0.095 ft-lb. per in. of notch will break none of the specimens completely but may crack some of them if above 0.083 ft-lb. per in. of notch (*C* in Fig. 2).
4. Shocks of greater than 0.16 ft-lb. per in. of notch will completely break all of the specimens (*D* in Fig. 2).
5. Shocks of 0.106 ft-lb. per in. of notch will crack half of the specimens (*E* in Fig. 2). This value may be considered to be the statistical mean energy-to-fracture.
6. Shocks of 0.142 ft-lb. per in. of notch will completely break half of the specimens (*F* in Fig. 2). This may be considered to be the statistical mean energy-to-break.
7. The difference between energy-to-break and energy-to-fracture, in this case 0.036 ft-lb. per in. of notch, is probably a measure of the energy consumed in completing the fracture, that is, tearing off the broken piece.
8. The difference between the shock which will fracture none of the specimens (*A*) and that which will fracture all of them (*B*), or between the shock which will break none of the specimens (*C*) and that which will break all of them (*D*) is an indication of the homogeneity of the test material. If this difference is large, that is, the values have a high degree of scatter, the material is relatively nonhomogeneous, and *vice versa.*

The statistical method is not convenient for routine testing, because it is relatively time-consuming and requires a large number of samples. Hence, two other tests to measure the energy required to just break a specimen were devised. The first of these, the small-excess-swing test, resembles the standard A.S.T.M. impact test except that initial hammer elevations are so chosen that the kinetic energy at impact is barely in excess of that required to break the specimen. The small amount of "follow-through" of the hammer is noted and this residual energy is subtracted from the initial potential energy setting to give the energy consumed in breaking the specimen. The value includes the energy required to initiate fracture, and to tear the specimen in two, but minimizes losses due to thrown fragments.

In the second test, the increasing blow method, the specimen is struck from an energy level slightly below that required to fracture, and the process is repeated from levels increasing by about 10 per cent until fracture occurs. Values by this method are usually slightly lower than those of the statistical energy-to-fracture method because of fatigue effects. (Both energy-to-fracture and energy-to-break values, as determined by the statistical method, are free from any fatigue effects.)

An estimate of the energy required to throw the broken specimen in the standard Izod test was obtained by replacing the fragments and noting the energy consumed in kicking them away by a second blow of the pendulum. This estimate may be inaccurate, as pointed out by Zinzow (**8**) and Werring (**9**), where the coefficients of restitution of the materials being studied differ appreciably, but for our tests the results seemed to be reasonably correct.

TABLE I.—IMPACT RESULTS BY SEVERAL METHODS.
All energies in foot-pounds per inch of notch.

Test Method			Molded Phenolic Compositions			Polystyrene		Polymethylmethacrylate, Cast
			Wood Flour Filler	Asbestos Filler	Chopped Canvas Filler	Injection-Molded	Cast	
Standard Izod	Total energy		0.263 ±0.012	0.265 ±0.007	2.94 ±0.24	0.305 ±0.070	... ...	... ...
	Energy to throw broken end		0.190 ±0.004	0.235 ±0.006	0.139 ±0.008	0.152 ±0.008	...	...
Statistical Method	To crack	None	0.083	0.050	0.85	0.13	0.100	0.115
		Half	0.105	0.059	1.07	0.18	0.165	0.133
		All	0.124	0.070	1.70	0.25	0.250	0.157
	To break	None	0.095	0.065	....	0.25	0.100	0.115
		Half	0.143	0.074	....	0.31	0.165	0.133
		All	0.155	0.110	....	0.35	0.250	0.157
Increasing-blow method			0.099 ±0.004	0.060 ±0.003	1.419 ±0.057	0.44 (approx.)	...	...
Small-excess-swing method			0.134 ±0.005	0.082 ±0.006	2.77 ±0.35	0.227 ±0.05	...	0.147 ±0.009

EXPERIMENTAL

Four molded phenolic compositions (containing wood flour, chopped canvas, cotton cord, and a mixture of asbestos and wood-flour fillers, respectively), two polystyrenes (one cast and one injection-molded), and cast polymethylmethacrylate were studied. Standard 0.5 by 0.5-in. bars, notched by milling (D 256) were used. The phenolics were preconditioned at 50 C. for 48 hr. and stored in a desiccator until tested (type S of the A.S.T.M. Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 41 T)).[4] The polystyrene and polymethylmethacrylate samples were conditioned and tested at 25 C. and 50 per cent relative humidity (A.S.T.M. D 618, type R).

Figure 2 shows the results for the wood-flour-filled phenolic. Similar curves were prepared for the other materials tested and the data obtained therefrom are summarized in Table I. Figure 3 shows the results for asbestos-filled phenolic, cast and injection-molded polystyrene, and cast polymethylmethacrylate. Results for fabric-filled phenolic are shown in Fig. 4. It will be noted that the various data on energy to fracture correlate much better with toughness of the materials, as deduced from practical experience, than do the standard Izod data. Specifically, the asbestos-filled formulation is shown to be more brittle than that containing wood flour, as is known from empirical experience to be the case. The effect of density of the specimen, which adds so materially to the "broken-end" error, is eliminated. Similarly, the comparative strength of fabric-filled and wood-flour-filled formulations more nearly approach those which service experience would lead us to expect, as do those of polystyrene and wood-flour-filled phenolic. The comparative homogeneities of these materials, estimated as outlined above, are also worth noting.

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[4] 1942 Book of A.S.T.M. Standards, Part III, p. 1053.

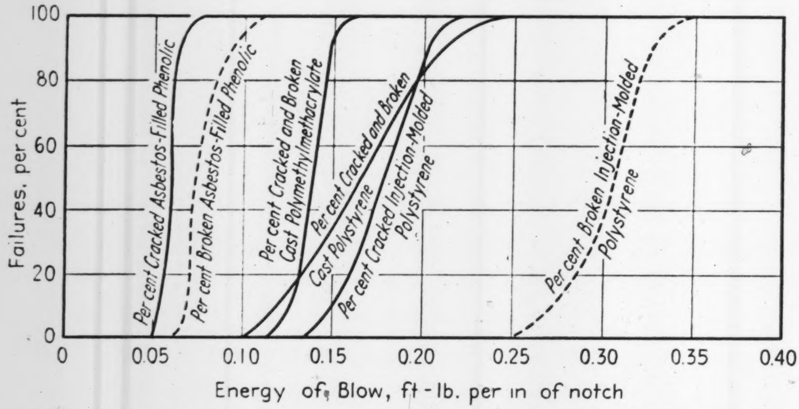

FIG. 3.—Effect of Single-Impact Blow on Standard Izod Specimen.

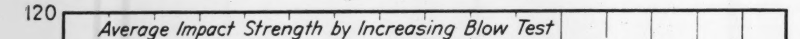

FIG. 4.—Effect of Single-Impact Blow on Standard Izod Specimen Fabric-Filled Phenolic Plastic.

Estimation of the energy to fracture has proven especially valuable during an investigation of the effect of continued heating on the strength of thermosetting plastics, and enabled several puzzling effects to be resolved rationally. For

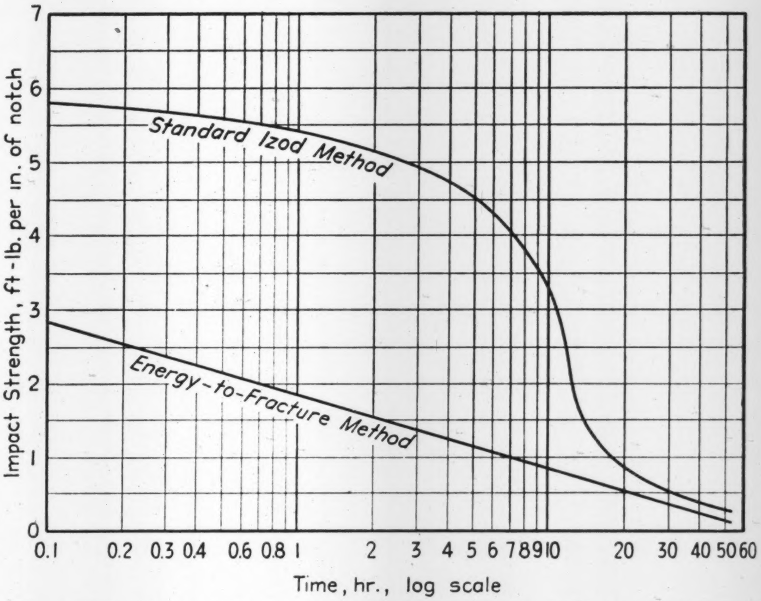

FIG. 5.—Impact Strength of Cotton-Cord Filled Phenolic Plastic Heated at 200 C. (392 F.).

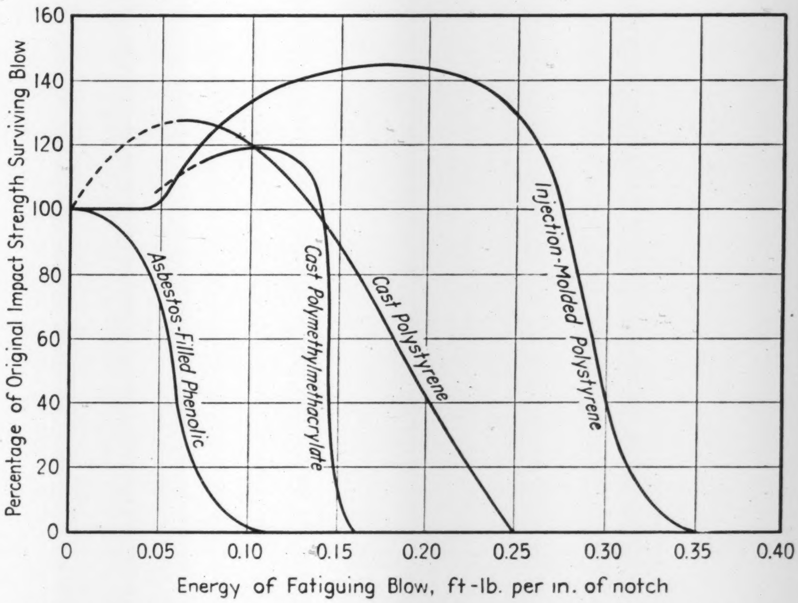

FIG. 6.—Effect of Single Fatiguing Blow on Impact Strength (Small-Excess-Swing Method).

example, Fig. 5 shows the effect of continued heating at 200 C. (390 F.) on the impact strength, as measured by the standard Izod test and by the "increasing blow" energy-to-fracture test, of a phenolic resin composition containing cotton cord as a filler. It will be noted that the resistance to cracking decreases exponentially with time of heating, whereas the resistance to breaking in the standard Izod test is complicated by the fact that the energy used in tearing shows an initial resistance to heat but breaks down with increasing speed as heating continues. Both values finally approach the strength of resin alone, as the reinforcing action of the cellulosic filler is destroyed by thermal decomposition.

The fatigue effect of single or multiple impact blows whose force is insufficient to fracture the test specimen can be estimated by another modification of this test. In this, specimens are struck by a blow (or blows) of any desired energy content and are then broken by any of the proposed methods (the small-excess-swing procedure is especially suitable). The residual strength of the specimen, after the fatiguing impacts, is then plotted, in percentage of the strength of similar specimens broken in a single blow, as a function of the energy level from which the fatiguing blow was struck. Figure 6 shows such data for an asbestos-filled phenolic composition, for cast and injection-molded polystyrene, and for cast polymethylmethacrylate, using the small-excess-swing test, and Fig. 4 shows similar information for a fabric-filled composition obtained by the standard Izod method. Single fatigue blows were used in both cases.

The data for cast and injection-molded polystyrene, and for cast polymethylmethacrylate are remarkable in that they show a negative fatigue effect, similar to the work-hardening of metals, when struck by blows whose energy is less than 80 per cent of that required to cause fracture. It may be that blows of lesser intensity cause enough plastic flow at the base of the notch to reduce the stress concentration factor during the subsequent fatal impact.

CONCLUSIONS

The results outlined above are, of course, limited in scope. They do seem to be sufficiently interesting, however, to justify further investigation of tests of this type. In the method we have employed, the velocity of the impact hammer must vary as the kinetic energy is varied. This is undesirable since the importance of fixing the velocity and varying only the available energy is known (**1**, **5**, **7**, **13**, **14**, **15**). The falling-weight method employed by Callender (**5**) and by Church and Daynes (**7**) offers a promising solution to this problem, and should be investigated in detail, using a greater number of types of plastics. Fatigue effects should be evaluated, using many blows of low energy content (Callender's ingenious device for insuring uniform speed with such low-energy impacts should be of value for this purpose). Callender's method for relating impact strength to specimen cross-section appears to be of great practical importance and should be studied in detail.

The intensive investigation of these points is recommended for the attention of the Society's Committee D-20 on Plastics.

Acknowledgment:

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DISCUSSION

Mr. I. L. Hopkins[1] (*presented in written form*).—Questions regarding the validity of the Izod test have been raised previously by Callendar, as well as by the authors of the present paper.

It is true that there has been much confusion in the data obtained with specimens of thermoplastics, and considerable research work on the impact strength of these materials is being done and much more is required. However, the Izod test has been very successful in controlling quality of phenolics and hard rubber, giving reproducible and significant results when certain factors are controlled. It should be mentioned that it is with materials which seem to be easily controlled in this way that Callendar, and Telfair and Nason, have done the work they have reported in their recent papers. It would appear to us that these papers do not consider two important points and are possibly in error in a third, provided they intend their conclusions to have general validity.

The first of these points has to do with the specimen itself. Callendar mentions cutting the finished product to obtain specimens for the impact test. This is a valuable and necessary procedure, but is of more use in evaluating the fabrication, for example molding, than purely as a materials test. In the manufacture of certain telephone parts, we have used an Izod test requirement on molded test bars as an acceptance test for the raw material, followed by a test on the molded parts themselves as an evaluation of the molding. We found that if the molding powder met the Izod requirement, the parts could be made to meet their impact requirements provided the molding were properly done. The accompanying Fig. 1 shows a diagram of an injection molded cellulose acetate or acetobutyrate combined telephone set mounting. There are 8 places where Izod specimens may be obtained from this mounting, as shown. The accompanying Table I gives the results of

TABLE I.—IZOD IMPACT STRENGTH OF SECTIONS OF A COMBINED TELEPHONE SET MOUNTING.

Specimens notched, conditioned at 120 F. for 1 day, cooled in desiccator.

Specimen	Impact, ft-lb. per 0.1 in. of notch		
	Min.	Avg.	Max.
No. 1	0.220	0.266	0.289
No. 2	0.258	0.299	0.321
No. 3	0.248	0.262	0.273
No. 4	0.198	0.249	0.287
No. 5	0.270	0.308	0.355
No. 6	0.067	0.083	0.098
No. 7	0.069	0.072	0.077
No. 8	0.065	0.078	0.100

Izod strength of regular injection molded test bars 0.107 ft-lb. per 0.1 in. of notch.

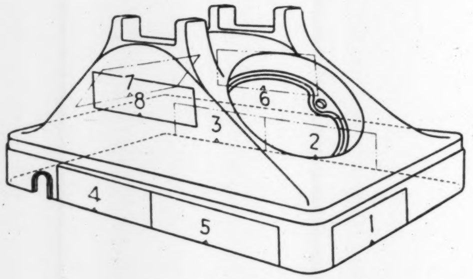

Fig. 1.—Injection Molded Combined Telephone Set.

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[1] Bell Telephone Laboratories, Inc., New York, N. Y.

impact tests on the specimens, each average being the result of approximately 15 specimens. The No. 7 specimens are the weakest, ranging from 0.069 to 0.077 ft-lb., averaging 0.072. The No. 5 are the strongest, ranging from 0.270 to 0.355 ft-lb. and averaging 0.308, or more than 4 times as much as the No. 7 specimens. This is very instructive as a survey of the points of strength and weakness of the mounting, but indicates that great care must be used in selection of the location from which to cut test specimens if one is to use them for evaluation of the material, and the question whether any intrinsic property of the material except its sensitivity to molding conditions is demonstrated is not easy to settle.

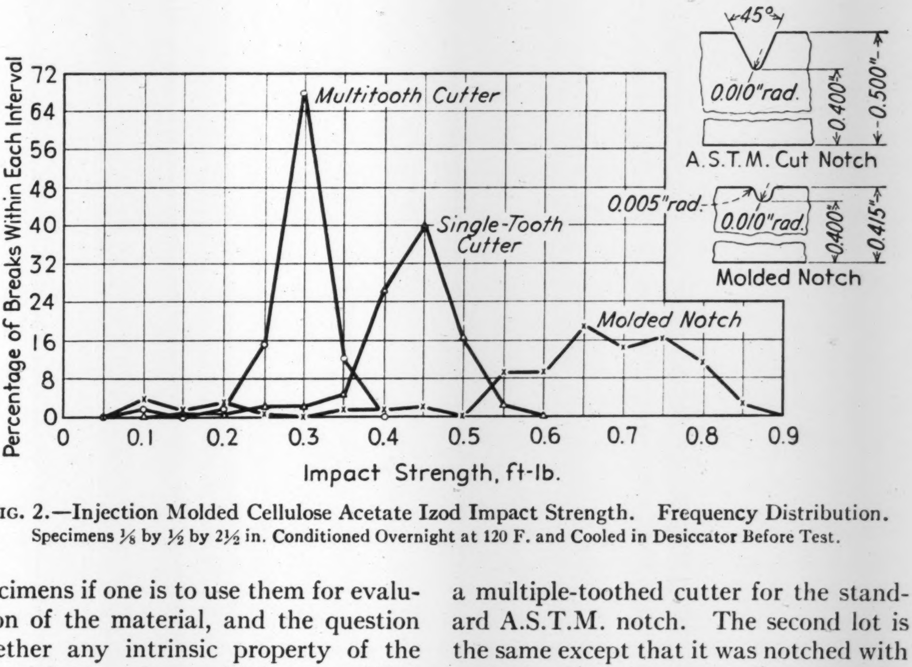

FIG. 2.—Injection Molded Cellulose Acetate Izod Impact Strength. Frequency Distribution. Specimens ⅛ by ½ by 2½ in. Conditioned Overnight at 120 F. and Cooled in Desiccator Before Test.

Another point of dissimilarity between the phenolics and hard rubber on one hand, and acetates and acetobutyrates, for example, on the other, is sensitivity to notching. It has been found, at the Bell Telephone Laboratories, that notching is adequately controlled for the phenolics and hard rubber if the cutter is sharp, conforms in contour to the specification within reasonable limits, and if ordinary care is used to have suitable feeds and speeds in milling. This is not so with acetates and acetobutyrates. Duplication of results is difficult and cannot yet be done with certainty in spite of the fact that a great deal of work has been done upon this problem. The accompanying Fig. 2 shows frequency distribution curves of Izod breaks made on specimens with three different notches. The first lot was notched with a multiple-toothed cutter for the standard A.S.T.M. notch. The second lot is the same except that it was notched with a single-toothed cutter. The third lot has a molded notch to test whether or not the spread of impact readings about the mean could be improved. In order to minimize the strains introduced by the flow of the material around the notch during the injection, the width of the bar was reduced from 0.500 to 0.415 in., and the notch was reduced to a depth of 0.015 in. The 0.010-in. radius and the 0.400-in. dimension beneath the notch were maintained. The results with the single-toothed cutter are about 50 per cent stronger than those with the multiple-toothed cutter. Those with the molded notch are over twice as strong and the spread is greater. This is not a solitary example but is merely part of a large collection of data demonstrating that such control as we now have is not good enough. It is offered to show that the notch should not be taken for granted as a settled problem to be used without question as a basis for other work.

TABLE II.—BALL DROP TEST ON ACETOBUTYRATE.

Each specimen subjected to one blow from each ball between the "at start" and "at break" balls inclusive, except for the last column where the same ball was used throughout any given test. Standard notched Izod specimens of acetobutyrate, injection molded ⅛ in. thick. Not conditioned, tested at 77 F.

At Start			At Break				Number of Blows to Break Using Same Ball as at Start
Ball Number	Height, in.	ft-lb.	Ball Number	Height, in.	Average, ft-lb.	Average, Total Number of Blows	
1	24	0.037	8-8-8	24	0.243	8	52
2		0.052	8-8-8		0.243	7	33
3		0.072	8-8-8		0.243	6	19
4		0.096	7-8-10		0.265	5.3	12
5		0.124	8-8-9		0.260	4.3	9
6		0.158	9-9-9		0.295	4	6
7		0.198	9-10-10		0.334	3.7	9
8		0.243	10-10-11		0.379	3.3	7
9		0.295	11-11-12		0.445	3.3	5
10		0.354	12-12-12		0.494	3	4
11		0.420	13-13-13		0.576	3	4
12		0.494	14-14-14		0.667	3	3
13		0.576	14-15-15		0.734	2.7	3
14		0.667	15-15-15		0.767	2	3
15		0.767	16-16-16		0.876	2	2
16		0.876	17-17-17		0.996	2	2
17		0.996	17(24") 17(26") 17(26")		1.022	1.7	2
17	26	1.035	17(26") 17(26") 17(27")		1.048	1.3	2
17	27	1.075	17	27	1.075	1	1

The third factor is fatigue. It appears that the materials discussed by Callendar and by Telfair and Nason have rather fortunate characteristics in this respect. Table II shows the results of some tests on notched cellulose acetobutyrate. These are ⅛ in. thick injection molded specimens tested in the ball drop machine with 24 in. drop and 0.866 in. distance from vise to striking point, except for the two largest balls whose diameters are so great that the distance must be increased somewhat. This table shows that if we start with the No. 6 ball, we will obtain breaks with the No. 9 ball at 0.295 ft-lb., after a total of 4 blows. If, however, we start with the No. 16 ball with an energy of 0.876 ft-lb., we obtain breaks with the No. 17 ball at 0.996 ft-lb., after a total of 2 blows. Either result would appear reasonable enough in the absence of the other, but taken together they indicate that fatigue effects are present to such degree as to mask completely the single blow strength. This indicates that care must be used in any increasing blow test method for evaluating single blow strength. Another thing brought out in this test is the time factor. To economize on time, all the samples to be struck with a given ball were struck by it in turn, the chute adjusted to give the 0.866 in. distance for the next size ball, and then all the specimens to be struck by this ball were struck in turn. Each specimen therefore had to wait its turn to be struck with any given ball and there was some delay between the blows given each specimen. A regular fatigue test was also run on the same material. In this test, a specimen was put in the machine and a given ball was dropped successively as rapidly as possible until the specimen broke. With the No. 8 ball, for example, it took 7 blows to break the specimen. When we started with the No. 3 ball, however, and worked up to the No. 8 ball, the specimens broke with a total of only 6 blows. This would indicate that the effect is more than merely cumulative, which can be accounted for only by assuming that the effect of a blow is increased as time elapses.

MR. H. H. BASHORE.[2]—I too should like to question the method of recording energy. For instance, in one application particularly that I have worked on, the highest impacts yielded the worst product in the final analysis in the product and the material that finally developed the best performance was the material which returned the highest amount of energy to its source. After we discovered this we dropped the pendulum from a point slightly (5 per cent) below the height required to break the specimen and then measured the rebound or energy returned. The results checked with actual conditions of service.

Have the authors done any work along these lines to show the difference which might exist?

If so, how do the results compare to the results obtained by observing the cracking point?

MR. DAVID TELFAIR.[3]—If I understand Mr. Bashore's question correctly, we have tried dropping the pendulum at energies below the energy to fracture value in the increasing-blow method. For blows of such energy the specimen deforms elastically and returns most of its energy to the pendulum. When a blow of just enough energy to crack the specimen is used, however, there is little or no rebound of the pendulum. Thus, the maximum energy of the rebound which he measured may be expected to be approximately the same as or perhaps a little lower than that required to fracture the specimens which we have measured.

For the materials we tried, which are not very representative, we found that the energy obtained by this method was only slightly lower than the energy needed to crack a specimen by the statistical energy-to-fracture method. But in some materials, as Mr. Hopkins points out, the fatigue effect is very marked, and it might be considerably lower for such materials.

MR. W. N. FINDLEY.[4]—There are several factors suspected of influencing the ability of a material to resist being fractured by a single impact. These factors are the magnitude of the stress developed at the most vulnerable spot during the impact, the temperature, the moisture content, the state of stress $\left(\text{the ratio of maximum normal stress to maximum shearing stress, } \frac{\sigma_{\max}}{\tau_{\max}}\right)$, the stress gradient $\left(\text{the slope of the stress distribution curve, } \frac{d\sigma}{dx}\right)$, and the rate of strain at the most vulnerable spot.

In the tests conducted by the authors the object was to maintain all influencing factors constant except one, namely, the magnitude of the stress, and to vary the magnitude of stress developed (by changing the energy of the blow) until the amount of energy required to produce fracture was determined.

It is, however, very difficult if not impossible to keep all the above factors the same in different specimens. This undoubtedly accounts in part for the fact that identical results are seldom obtained from similar specimens. The last two factors, stress gradient and rate of strain are perhaps the most difficult to control. The stress gradient may be altered by residual stresses built up during molding or machining. However, the importance of the stress gradient as a factor in impact resistance is yet to be established.

The effect of rate of strain is, however, known to be important and it is unfortunate that the authors were not able to devise a method of conducting their tests in which the rate of strain could be kept constant. The effect of change in rate of strain may be small except in the vicinity of a critical condition, corresponding to certain values of the influencing factors—temperature, moisture content, state of stress, stress gradient, and rate of strain. At the critical condition a small change in any one of these factors may cause a shift in the mode of failure of the material from ductile to brittle, or *vice versa* with a resulting marked change in the energy which a specimen can absorb without rupture.

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Two possible explanations for the apparent increase in impact strength of thermoplastics resulting from a "fatiguing" blow (Fig. 6) are as follows:

1. If the fracturing blow followed the fatiguing blow immediately, there may have been a considerable rise of temperature in the specimen. Such a rise in temperature may have led to a greater energy absorption because of the larger plastic flow which would have accompanied a higher temperature.

2. The fatiguing blow may have left a residual stress distribution such that the material at the root of the notch was under a compressive stress. Thus, on the fracturing blow the specimen must be deformed farther in order to overcome the effect of the compressive residual stress.

MR. BERNARD EPSTEIN.[5]—In order to avoid the difficulty involved in determining the first appearance of cracks in the specimen, it is suggested that a light colored glossy finish might be used to advantage. The coating, however, must not be too elastic or too brittle. If the finish is too flexible, the coating might not show cracks, although the specimen itself might be cracked. If the coating is too brittle, cracks may appear in the finish before they appear in the test specimen.

On occasion we have made use of the standard Navy M-485a light gray nonspecular lacquer for such purposes. Although no attempts were made to determine the relative brittleness of the coating, it was a definite help in determining the first appearance of cracks. It was also apparent, however, that a glossy finish would have been even more helpful. The test in which the lacquer was used was a falling ball test on a black phenolic part.

Although the point raised by Mr. Findley concerning the effect of rate of loading is certainly valid, the error introduced by this factor is believed to be small.

An uncontrolled rate of loading might be expected to introduce errors; however, the order of magnitude of such errors are thought to be much smaller than the errors introduced by the energy absorbed in tearing the specimen apart and kicking it across the room.

Although no complete and formal attempts have been made by us to determine the effects of rates of loading, the results of one set of tests tends to indicate that the effect of rate of loading is not as great as might first be expected. The test was basically an attempt to determine the material to use for a part which was being subjected to the impact of cartridge cases ejected into the slip stream on the underside of a wing. Within the crudity of the test, the results appeared to be independent of rate of loading.

MESSRS. DAVID TELFAIR and HOWARD K. NASON[6] (*authors' closure, by letter*).—Mr. Hopkins' comments are interesting and instructive. His use of the standard Izod test for the evaluation of both raw material and molding technique is an excellent example of the potential usefulness of impact testing. Certainly, the standard test distinguishes between materials and methods of fabrication in a way which for many types of materials is very useful. We have attempted to point out, however, that the standard test does not measure a definite physical property. The test values are representative, not of the energy required to fracture the specimen, but of the sum of several energies, some of which have little or no relation to brittleness. We have pointed out that for at least two relatively important types of materials the presence of these unwanted energies tend to obscure the desired quantity. We are, therefore, convinced that any impact test which more nearly measures the true energy to fracture the specimen would be of increased value.

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[5] Chance Vought Aircraft Division of United Aircraft Corp., Stratford, Conn.

[6] Assistant Director of Research, Plastics Division, Monsanto Chemical Co., Springfield, Mass.

We are glad that Mr. Hopkins has pointed out the difficulties in connection with notch effects and fatigue effects. We agree that these are important and that they should be investigated more completely.

We are convinced that any tests which are representative of a combination of singly simple properties should, in so far as possible, be avoided. In other words, notch sensitivity, impact fatigue, and single blow impact should each be written up eventually as separate tests. For this reason we have limited our present paper to a discussion of the standard Izod method, a single blow impact test.

Mr. Findley's comments on the effect of rate of strain are pertinent. The authors regret that they were unable to keep this factor constant in their testing. However, as Mr. Epstein has just pointed out, the effect of rate of strain is quite small for all the materials tested by us (except perhaps cellulose acetate). Our highest impact velocities were certainly not more than 2 or 3 times as great as our lowest. On the other hand, the rate of straining was of the order of magnitude of 20,000 times as great as that used in static flexural testing. The energy to crack a notched specimen of phenolic plastic in static flexure, as measured by us, is only slightly less than the energy to crack the specimen in impact, as determined by the statistical E. T. F. method.

THE RELATION BETWEEN RESULTS OF REPEATED BLOW IMPACT TESTS AND OF FATIGUE TESTS

By William N. Findley[1] and Otto E. Hintz, Jr.[2]

Synopsis

Results of repeated blow impact tests on cellulose acetate were compared with results of fatigue tests of the same material by plotting the stress *versus* the number of cycles (or blows) for failure. Calculation of the maximum stress resulting from the impact is discussed. It was observed that failure resulted from progressive fracture for both types of tests, and that the data for the stress and number of cycles, when plotted, showed close agreement between the two types of tests. This agreement indicates that the phenomenon of fracture after repeated impact is the same as that resulting from fatigue.

Purpose

The tests reported in this paper were made to determine the effect of repeated-blow impact loading on cellulose acetate, and to evaluate this effect in terms of the fatigue properties of the material. This project was undertaken because there are many instances of repeated impact loading which occur in service.

The effect on plastics of repeated impacts has been studied by several investigators[3, 4, 5] as has the general subject of repeated impact on materials.[6] However, much of the work that has been done to date has not been published. Some of these investigators found that failures occur in plastics after a number of repetitions of bending impact in which the energy delivered to the specimen per blow was less than 10 per cent of the energy required to fracture the specimen in a single blow. Some investigators concluded that a limiting value of energy was reached after about 100 to 300 blows, below which value fracture would not occur even with an indefinitely large number of blows.

In order to study the relation between repeated impact tests and fatigue tests a shape of specimen was chosen which would allow calculation of the stress at the point at which fracture started. Both repeated impact tests and fatigue tests were performed with this specimen and the stress and number of cycles for fracture were determined for each type of test.

Material and Specimens

The cellulose acetate plastic used for these tests was supplied by the Plastics

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[2] Radio Corporation of America, Camden, N. J., formerly student in the College of Engineering, University of Illinois.

[3] R. Burns and R. W. Blackmore, "Impact Fatigue Endurance Limit of Plastics," Experimental Records of Bell Telephone Laboratories, Inc., received by W. N. Findley in private communication with the author, 1942.

[4] L. H. Callendar, "New Methods for Mechanical Testing of Plastics," *British Plastics*, Vol. 13, Part I, No. 155, April, 1942, p. 445; Part II, No. 156, May, 1942, p. 506.

[5] Report of Committee D-9 on Electrical Insulating Materials, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 399 (1933).

[6] S. Timoshenko and J. M. Lessells, "Applied Elasticity," Westinghouse Technical Night School Press, p. 434 (1925).

Division of the Monsanto Chemical Co. The material was a clear, transparent thermoplastic composed of medium viscosity cellulose acetate of the acetone soluble type, plasticized with about 26 per cent of phthalate and aromatic phosphate ester plasticizers. The Monsanto formulation number was 2050 T.V. This formulation is the same, except for slight change in plasticizer, as that for which test results were reported in three previous papers by the senior author.[7, 8, 9]

All specimens used in these tests were cut from the same sheet of cellulose acetate plastic, but not the same sheet as used for the previously reported tests. The sheet was 0.3 in. thick and was made by the sheeter process at a molding temperature between 200 and 250 F. The finished sheet contained less than one per cent of residual solvent and water.

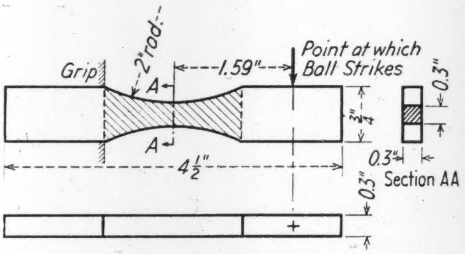

FIG. 1.—Specimen.

The specimen used is shown in Fig. 1. The bending impact and fatigue specimens were formed by sawing rectangular blocks from the sheet, and milling the sides and the reduced section. During all machining operations care was used to avoid heating the specimens, as this might change the properties. All machining was done in a room maintained at a constant temperature of 77 F. and constant relative humidity of 50 per cent. This was done to avoid exposing the material, which had been in the laboratory for about two months, to a different atmosphere. All tests were also conducted in this same room.

All specimens were hand polished over the reduced section to give a uniformly smooth surface so as to avoid stress concentration. Three grades of paper, numbers 0, 00, and 000 were used successively, and polishing was done slowly enough to avoid heating.

MACHINES AND METHODS OF TESTING

Repeated Impact Machine:

The impact test was performed by causing a steel ball to fall from a fixed height and strike the end of a specimen mounted as a cantilever beam. The height of drop used in this series of tests was 22.6 in. This height produced a velocity at the time the ball struck the specimen of 11.0 ft. per sec. (the striking velocity used in the "standard" impact test).[10]

Tests which required less than 1000 blows to cause fracture of the specimens were performed by hand. When a larger number of blows was required, apparatus was provided to repeat the blows automatically at a rate of about 100 blows per min.

The equipment used for the repeated impact tests is shown in Fig. 2. The specimen, *A* in Fig. 2, is clamped, as shown in Fig. 3, to a heavy steel rail imbedded in a concrete block. This massive support was used in order to reduce to a minimum the energy adsorbed in deflection of the support.

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[7] William N. Findley, "Mechanical Tests of Cellulose Acetate," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 1231 (1941).

[8] William N. Findley, "Mechanical Tests of Cellulose Acetate—Part II on Creep," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 914 (1942).

[9] William N. Findley, "Mechanical Tests of Cellulose Acetate, Part III," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 65, No. 5, p. 479 (1943); *Modern Plastics*, Vol. 20, May, 1943, p. 99.

[10] A.S.T.M. Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 - 41 T), 1942 Book of A.S.T.M. Standards, Part III, p. 1071.

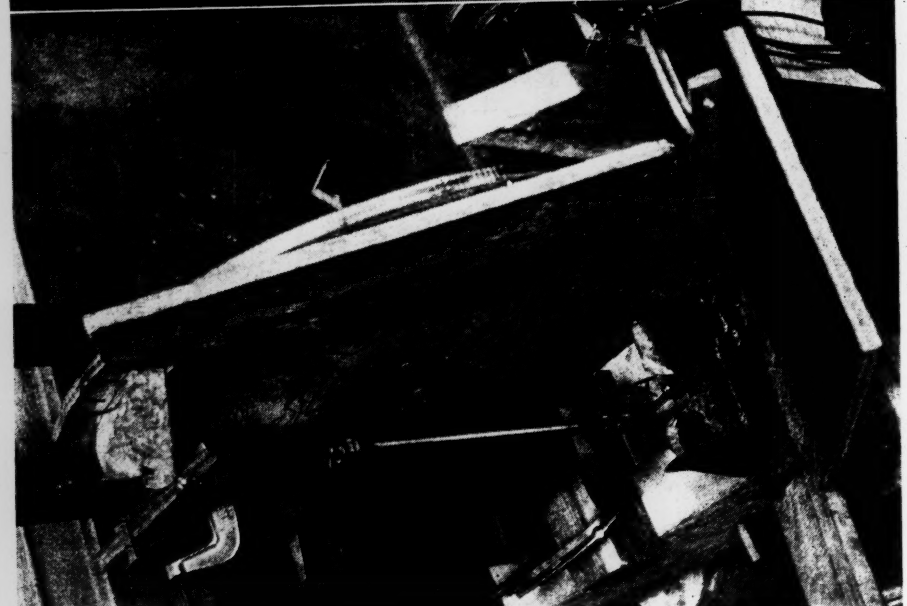

FIG. 2.—Repeated Impact Machine.

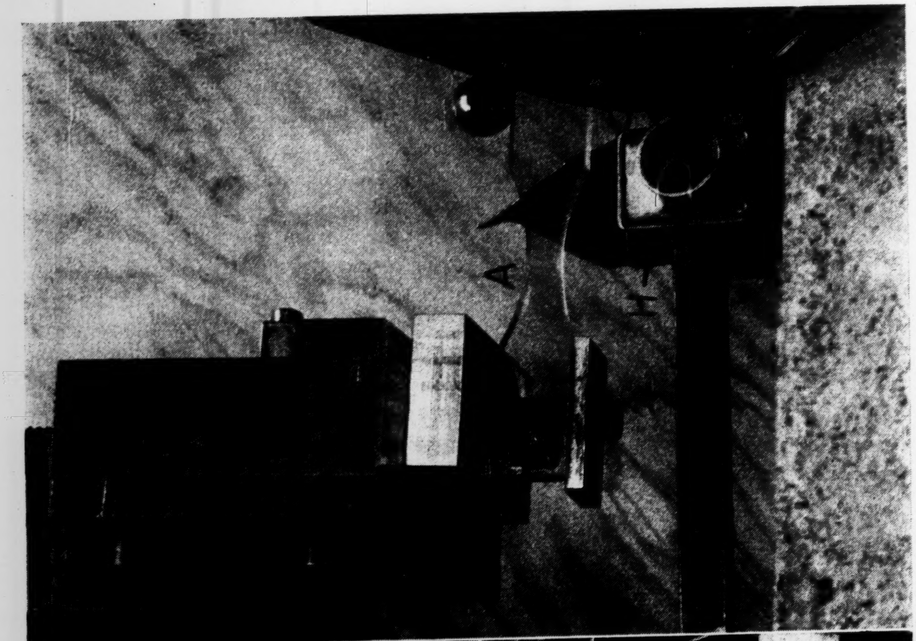

FIG. 3.—Detail of Specimen in Impact Machine.

Steel balls were caused to roll off the end of a trough having a gentle incline (*B*, Fig. 2). The trough was adjusted by sliding forward or back to the position which would cause the falling ball to strike at the center of the flat top face of the specimen (see Fig. 3). When the apparatus was operated by hand the ball was caught on rebound from the specimen and returned to the trough *B* by hand. For automatic operation several balls were used and a motor driven elevator was provided to return the balls to the trough *B* from which they fell.

This elevator consisted of a large wheel (*C*, Fig. 2) containing several pockets spaced around its periphery. The wheel was driven at slow speed by a motor. As each pocket *D* passed the hopper *E* containing the balls to be returned, a ball entered the pocket and was carried up by the wheel and discharged at *F*. From this point the ball entered the trough *B*, rolled slowly down to the end of the trough, and repeated the impact operation.

For automatic operation, a counter (*G*, Fig. 2) was provided to count the number of revolutions of the wheel *C*, and a micro-switch (*H*, Fig. 3) was used to stop the wheel when the specimen had fractured. The number of blows required to cause fracture was determined by multiplying the number of revolutions of the wheel by the number of pockets in the wheel.

In conducting a test, a specimen was fixed in the machine and then repeatedly struck by the same size ball until fracture occurred. Another specimen was then placed in the machine and the operation repeated using a smaller size ball. Balls ranging in size from 2 in. to $\frac{19}{32}$ in. in diameter were used. The energy delivered per blow corresponding to the different size balls ranged from 25.4 to 0.70 in-lb. respectively.

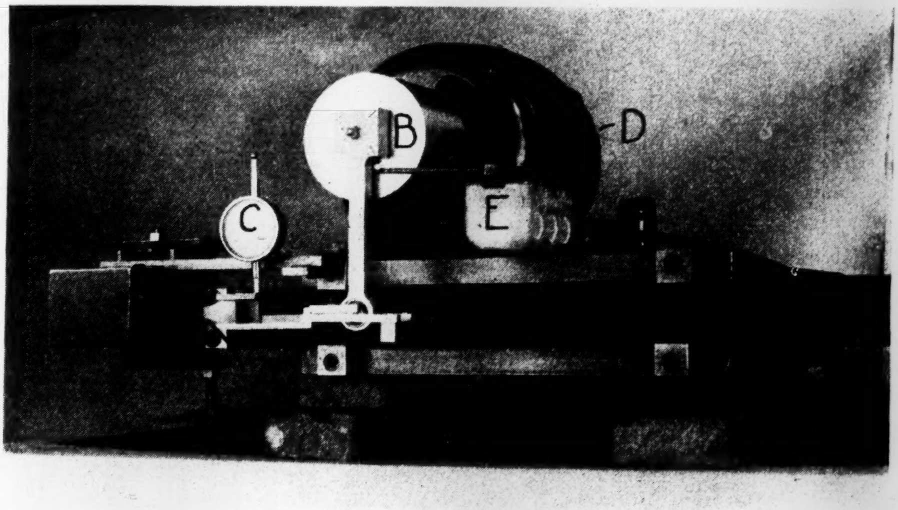

FIG. 4.—Fatigue Machine.

Fatigue Machine:

The fatigue tests were conducted on a fixed-cantilever, constant-amplitude fatigue machine. In this type of machine the specimen (*A*, Fig. 4) was repeatedly bent back and forth as a cantilever beam by the variable eccentric *B*. The procedure in conducting these tests was as described in the A.S.T.M. Tentative Method of Test for Repeated Flexural Stress (Fatigue) of Plastics (D 671 – 42 T).[11] The stress, σ, in the specimen (Fig. 1) at the minimum section was computed from the equation $\sigma = \frac{Mc}{I}$. The bending moment, M, was obtained by calibration of the specimen as a beam by means of dead weights. The deflection of the specimen under load was measured by means of a dial gage (*C*, Fig. 4). The number of cycles to which the specimen was subjected was recorded on a counter, *D*, and a toggle switch, *E*, was arranged to stop the machine when the specimen fractured. Thus for each specimen placed in the machine the stress corresponding to the deflection of the specimen during the test was calculated from the bending moment measured while the machine was at rest, and the number of cycles for fracture was obtained.

CALCULATION OF STRESS DUE TO IMPACT

From the principle of conservation of energy, derived from Newton's second law of motion, it is known that the potential energy (measured with respect to the bottom of the drop) which the ball had at the start of the descent was equal to the maximum strain energy produced by the impact in the specimen and support, plus the energy dissipated in heat resulting from solid-friction and dry-friction.

The potential energy of the ball was approximately equal to the product of the weight of the ball and the height of the end of the trough *B* (Fig. 2) above the surface of the specimen. Two other parts of the potential energy are small enough, compared to the total, to be neglected: (1) the potential energy equal to the product of the weight of the ball times the deflection of the specimen, and (2) the potential energy of the ball at the start of the descent with respect to the end of the trough. This part of the potential energy is transformed into kinetic energy of rotation and approximately horizontal translation due to the ball rolling down the inclined trough.

The maximum strain energy in the specimen occurs when the specimen reaches its maximum deflection. The strain energy may be expressed in terms of three components for this problem: strain energy of bending, strain energy of "horizontal" shear, and strain energy due to the localized compressive stress resulting from contact pressure between the ball and the specimen.

In order to evaluate the strain energy in bending and in shear in terms of the maximum contact force, P, it was assumed that the distribution of strain (and stress), in the specimen, resulting from the impact was the same as that which would result from a static load equal to P. This, of course, was not exactly true, but does not involve serious error since most of the strain took place near the minimum section of the specimen. Calculation of the strain energy in bending and shear to a first approximation indicated that the strain energy in horizontal shear was negligible compared to that of bending. The amount of strain energy due to compression was also neglected because of the fact that the stresses in compression were such that permanent deformation was not observed and the volume of material subjected to high stress under the contact was small compared to the volume of material subjected to high bending stresses.

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[11] 1942 Book of A.S.T.M. Standards, Part III, p. 1251.

The collision of the ball striking the specimen starts an elastic or sound wave which is propagated from the point of contact. This wave is reflected back and forth in the specimen in a complicated manner. The stress introduced in the specimen by this wave is probably not large, except in the region of contact, and is probably reduced nearly to zero as a result of damping by the time the specimen has reached its maximum deflection. The elastic wave will have been reflected back and forth several times during the period required for maximum deflection of the specimen.

Thus, if the energy transformed into heat and the strain energy stored in small quantities in horizontal shear, compression, etc., are neglected, the maximum stress at the minimum section can be calculated by equating the product of the weight of ball and the height of drop, to the strain energy of bending in the portion of the specimen cross hatched in Fig. 1.

The strain energy of bending in a beam may be calculated from the equation[12]

$$U = \frac{1}{2}\int_0^l \frac{M^2\,dx}{EI}$$

where:

U = the energy in inch pounds,

M = the bending moment in inch pounds,

E = the modulus of elasticity in pounds per square inch,

I = the moment of inertia of the cross-section in (in.)^4 and

x = the distance along the beam.

In the impact specimen used, the moment of inertia I changes with x and can be expressed as an equation of the following form

$$I = a(b - \sqrt{-x^2 + cx + d})^3$$

where a, b, c, and d are constants depending on the dimensions of the specimen. The bending moment M may be expressed in terms of the contact force of the ball as $M = P\,(x + e)$ where e defines the location of the point of contact of the ball. When these expressions were substituted in the equation for the strain energy, a rather complicated integral resulted:

$$U = \frac{P^2}{2Ea}\int_0^l \frac{(x + e)^2\,dx}{(b - \sqrt{-x^2 + cx + d})^3}$$

This integral was evaluated by the use of partial fractions and trigonometric substitutions. It was then possible to solve for the maximum contact force P in terms of the strain energy. Then, from the force P the maximum stress at the minimum section was computed using the flexure formula $\sigma = \frac{Mc}{I}$. The resulting equation was $\sigma_{\max} = 4.34\sqrt{\frac{UE}{w}}$

where:

$\sigma_{\max.}$ = the maximum stress at the minimum section of the specimen,

E = the modulus of elasticity in pounds per square inch,

w = the width of the specimen in inches,

U = the strain energy in inch pounds (which is replaced by the product of the weight of the ball times the height of fall), and

4.34 = a dimensional constant.

This constant applies only for a specimen of shape as shown in Fig. 1 when the point of contact of the ball is 1.59 in. from the minimum section of the specimen. Since the above equation involves the flexure formula it does not apply accurately to cases in which stress is not proportional to strain, as is the case when general yielding occurs. The modulus of elasticity of the material as determined from a tension test was 195,000 psi.

The above equation is not exact for

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[12] See any advanced book on strength of materials.

the reasons discussed above, and some others of lesser importance. It is believed, however, that the equation is sufficiently accurate to warrant the conclusions drawn below.

RESULTS

The data from both the repeated impact and the fatigue tests are plotted in Fig. 5. In this figure the maximum stress of the cycle for both impact and fatigue tests is plotted against the log of the number of cycles for fracture. The open circles represent data obtained from repeated impact tests, and the filled circles represent data obtained from fatigue tests. A progressive fracture was observed to occur at the minimum section of the specimen. This type of fracture occurred in both repeated impact and fatigue tests, and the appearance of the fracture was substantially the same in both cases.

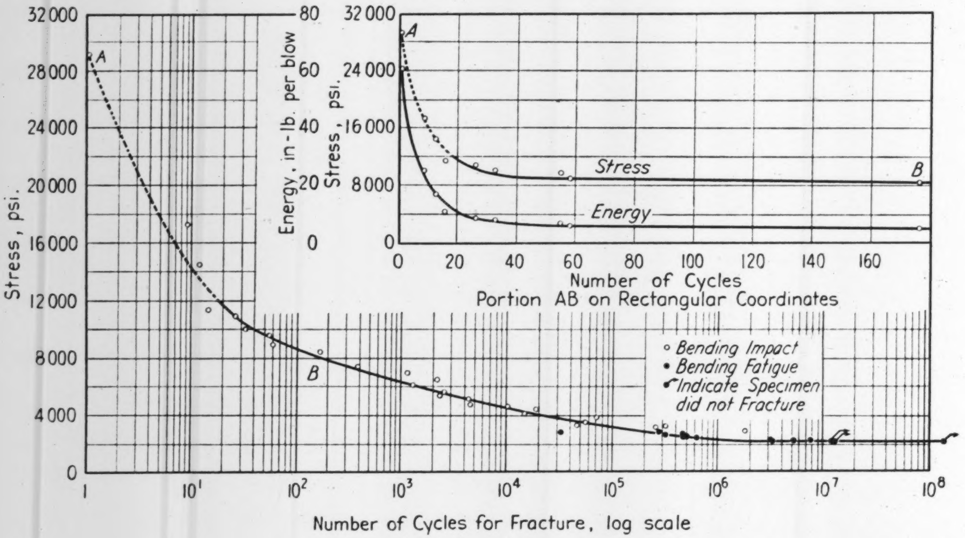

FIG. 5.—Stress *versus* Cycles (S-N) Diagram for Repeated Impact and Fatigue Tests of Cellulose Acetate in Bending.

For the fatigue tests shown, the range of stress was a complete reversal. That is, the specimen was bent up and down equally in each direction. However, in the repeated impact tests the range of stress was from zero to a maximum. Thus it was necessary to take account of the effect of range of stress in order to compare the fatigue tests with repeated impact tests. Previous fatigue tests of cellulose acetate at different ranges of stress[9] showed the relationship between the range of stress and the endurance limit of the material.

In order to compare the fatigue tests shown in Fig. 5 with the repeated impact tests, the fatigue test data were adjusted to correspond with a range of stress from zero to a maximum by multiplying the stress for each test by the ratio of the maximum stress of the cycle at the endurance limit for a range of stress from zero to a maximum, to the endurance limit for a completely reversed range of stress, obtained from the previously reported tests.[9] This ratio was 1.6.

The data shown in Fig. 5 were so adjusted.

Figure 5 shows that good agreement existed between the results plotted from the two different tests. That is, the data from the repeated impact and fatigue tests fell on one continuous curve. Thus the conclusion is drawn that the phenomenon of fracture resulting from repeated blow impact tests stems from the same cause as fatigue (fracture resulting from a crack which begins and progresses under the action of repeated loading). The character of the fractured surface and the growth of the crack were observed to be essentially the same for both types of test.

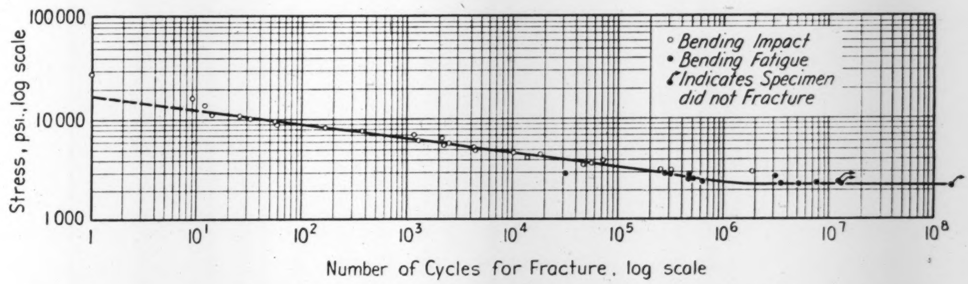

FIG. 6.—Stress *versus* Cycles (*S-N*) Diagram for Repeated Impact and Fatigue Tests of Cellulose Acetate in Bending, Double Log Plot.

Figure 5 shows that an endurance limit is reached at about one million cycles. This is evident from the fact that the curve becomes a horizontal line at one million cycles and remains horizontal out to about 200 million cycles (the duration of the tests).

The diagram inserted in Fig. 5 represents the same data up to 180 cycles (*A* to *B* on the large curve) plotted on rectangular coordinates. Also shown is the energy-per-blow plotted against the number of cycles (or blows). Either of these two curves would lead one to believe that an endurance limit was approached after about 40 cycles. Comparison with the semi-log plot, however, shows that the endurance limit is not reached until about one million cycles or a stress about one fourth that indicated by the rectangular plot. The energy per blow corresponding to the endurance limit was 0.38 in-lb. Comparing this with the energy required to fracture a specimen in a single blow, 61 in-lb., it was found that the energy at the endurance limit was less than one per cent of the single blow energy. (The single blow test was performed on a modified Izod impact machine because large balls were not available.)

Figure 6 shows the data of Fig. 5 plotted to a double log scale. It was found when thus plotted that the data fell along a straight line from about twelve blows to one million blows. This is the region in which fracture occurred without general yielding. General yielding was observed in all specimens which failed in less than twelve blows.

The portion of the curve, Fig. 5, from *A* to *B* does not accurately represent the stress in the tests shown because of the fact that general yielding preceded failure by progressive fracture in most of these specimens so that the stress in the specimen was not proportional to the strain and therefore the equation used in calculating the stress did not apply accurately.

The straight-line portion of the curve in which fracture occurred without general yielding can be represented by the following equation

$$\sigma = \frac{17{,}000}{N^{0.143}}$$

where: N is the number of cycles for fracture and σ the maximum stress of the cycle whose range is from zero to a maximum. The equation is good only for values of bending stress σ between 2160 psi. (the maximum stress of the endurance limit) and 11,000 psi. and provided the temperature remains low enough that failure does not occur due to overheating, and provided also that the rate of strain is high enough to prevent general yielding from occurring at stresses lower than 11,000 psi. The static tensile strength of this material was 4000 psi. at a rate of strain of 0.012 in. per in. per min.

Data were not obtained for fatigue tests at stresses higher than those shown because the heat which developed in the specimen due to internal friction at stresses well above the endurance limit caused failure by softening resulting from overheating rather than a failure by progressive fracture, so that higher stresses could not be used in the fatigue tests.

The time interval between impacts in the repeated impact test was long enough so that heating did not result. A temperature rise was however observed in all fatigue tests. This rise in temperature was from 10 to 100 F. depending on the stress used. Correction of the fatigue data for rise in temperature would make for even better agreement between the two types of tests since an increase in endurance limit with reduction in temperature was observed with cellulose acetate.[9] Correction of the fatigue test data to room temperature would increase the maximum stress at the endurance limit obtained from fatigue tests about 14 per cent to 2460 psi. instead of 2160 psi. for a range of stress from zero to a maximum.

After about 100,000 blows a crack was observed to form normal to the surface of the specimen at the point of contact with the striking ball. No indentation or marking of the surface of contact was observed to result from the striking of the balls until 100,000 blows. Tests with several different size balls indicated that the number of blows required to initiate this crack did not vary greatly with the size of ball.

CONCLUSIONS

From the results of fatigue tests and repeated impact tests of identical specimens conducted in a room maintained at constant temperature, 77 F., and constant relative humidity, 50 per cent, it was concluded that:

1. Failure under repeated impact resulted from progressive fracture the same as occurred in fatigue tests.
2. An endurance limit existed at a stress of 2160 psi. (the maximum stress in a cycle having a range from zero to a maximum). See Fig. 5.
3. The endurance limit was reached at about one million cycles. See Fig. 5.
4. The energy per blow corresponding to the endurance limit for a range of stress from zero to a maximum was less than one per cent of the energy required to fracture a specimen in a single blow.
5. General yielding occurred in specimens which fractured in about twelve blows or less.
6. The straight-line portion of the $\sigma - N$ curve (log log plot) in which fracture occurred without general yielding can be represented by the equation

$$\sigma = \frac{17{,}000}{N^{0.143}}.$$

This equation allows the calculation of the maximum stress due to impact which may be used in design of a part which will be subjected to a given number of impact stresses during its life.

7. The data shown in Fig. 5 seem to indicate that the endurance limit for repeated impact can be determined from fatigue tests.

8. A crack was also observed to occur normal to the surface of the specimen at the point of contact with the striking ball. This crack did not appear until about 100,000 blows, and did not result in fracture of the specimen.

Acknowledgments:

The tests reported in this paper were a part of the work of the Engineering Experiment Station of the University of Illinois (Dean M. L. Enger, director) in the Department of Theoretical and Applied Mechanics of which F. B. Seely is head. The authors are indebted to F. B. Seely for suggestions and criticisms during the preparation of this paper. The authors are also grateful to S. Yurenka and W. J. Worley, student test assistants, for their help on this project.

Acknowledgment is also made to the Plastics Division of the Monsanto Chemical Co. for the material supplied.

DISCUSSION

Mr. David Telfair.[1]—These are very interesting data to us, because we have been interested for some time in the relationship between the impact test and the flexure test. This paper confirms some of our results, at least in some measure. It seems to me to exemplify a highly commendable approach to many of the problems which are faced in connection with the testing of plastics, namely, that of analyzing and correlating various tests from the point of view of the fundamentals involved.

I should like to ask whether the authors have any information on the mean rate of stressing in repeated impact as compared with that in the flexural fatigue test.

Mr. William N. Findley.[2]—The mean rate of strain (and mean rate of stressing) was determined qualitatively from the duration of impact and was found to be of the same order of magnitude as the mean rate of strain used in the corresponding fatigue test.

Mr. T. Smith Taylor.[3]—I am extremely interested in the results presented in this paper because I worked a great deal with impact tests in the early days.

One thought that I raise is this: What attention has been given, if any, to the time of contact? In other words, in the case of a pendulum machine, what influence does the length of time that the fracture is being made have upon the results?

Some of the materials with which these investigators have been working will flex considerably during fracture. The time of contact during fracture is a reasonable percentage of the time which has elapsed during the time of fall of the pendulum, for example.

Mr. Findley.—It is known that for both metals and plastics the rate of strain influences the resistance of materials to fracture.[4,5,6,7,8,9] This is true both of impact tests, "static" tension tests, and fatigue tests. In the present investigation, however, the purpose was to keep the rate of strain as nearly constant as possible. The authors hope to extend the scope of the study to include a study of the effect of rate of strain.

Mr. C. R. Stock.[10]—We have gotten rather sketchy information on that subject which has no direct bearing on the thermoplastics that are under discussion, because the materials we investigated were rather stiff and, therefore, the deflection that took place could be expected to be rather small. But we were able to take high-speed photographs of the fracture that occurred in Izod impact.

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[2] Associate in Theoretical and Applied Mechanics, College of Engineering, University of Illinois, Urbana, Ill.

[3] Chief Engineer, Rectifier Division, Federal Telephone and Radio Corp., East Newark, N. J.

[4] L. H. Callendar, "New Methods for Mechanical Testing of Plastics," *British Plastics*, Vol. 13, Part I, No. 155, April, 1942, p. 445; Part II, No. 156, May, 1942, p. 506.

[5] William N. Findley, "Mechanical Tests of Cellulose Acetate," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 1231 (1941).

[6] William N. Findley, "Mechanical Tests of Cellulose Acetate, Part III" *Transactions*, Am. Soc. Mechanical Engrs., Vol. 65, No. 5, page 479, 1943; *Modern Plastics*, Vol. 20, May, 1943, p. 99.

[7] P. G. Jones and H. F. Moore, "Effect of Rate of Strain on Yield Strength, Tensile Strength, Elongation, and Reduction of Area in the Tension Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 488 (1941).

[8] William N. Findley, "Mechanical Properties of Macerated Phenolic Molding Material," National Advisory Committee for Aeronautics, *Advance Restricted Report No. 3F19*, June, 1943.

[9] C. Zener and J. H. Hollomon, "Plastic Flow and Rupture of Metals." Presented at the annual meeting of the American Society for Metals, October, 1943, *Preprint No. 9*. (This paper contains references to other work on the subject.)

[10] Physicist, American Cyanamid Co., Stamford, Conn.

The interval between photographic frames was approximately 1/1500 of a second and we found that that was too long a time because there was no fracture in one frame and the fracture was, as far as we could tell, complete in the second frame.

That seems to indicate that the thing goes rather rapidly, at least for the stiff materials. It may also hold for these materials since the ratio of the moduli of elasticity, which would have some bearing on the time for fracture, is in the neighborhood of, say, about 10 to 1. I should say the flexural strength of these materials was about 6000 psi. and the material we were testing was in the neighborhood of 12,000 psi. So you have a flexural strength ratio of about 2 to 1 and a modulus of elasticity ratio of about 10 to 1. Therefore, just roughly, you could say that the interval occupied between nonbreak and complete fracture would be somewhere in the neighborhood of two to ten times the interval of time that we observed.

MR. FINDLEY.—The author has also had a similar experience in connection with a photoelastic study of the photoelastic material (Bakelite BT61893) under high-speed bending.[11] A special camera capable of recording 3000 frames per second was used. It was observed that complete fracture occurred in the interval of time between succeeding frames.

MR. J. W. WESTWATER.[12]—What was the criterion of failure for the tests? Was it the energy to just fracture, energy to rupture, or perhaps the energy to cause a change in the modulus of elasticity? It occurred to me that, since the number of cycles was great, perhaps any of these measurements would give nearly the same result.

Secondly, was any difficulty encountered in getting the balls to hit the target? One might expect the balls to wobble somewhat while rolling down the trough.

MR. FINDLEY.—The criterion of failure which we used was complete separation of one end from the other. The quantity which was varied was the energy delivered in the blow, that is, the size of the ball (the height of the drop was the same). The quantity which was measured was the number of blows required to cause complete separation.

For very large energies which fractured the material in two to six blows there was no crack visible in the specimen just preceding the blow which completely fractured the specimen. But at lower energies the crack was observed to grow at a relatively slow rate depending upon the stress applied. The number of blows between the start of the crack and complete separation was, however, a small percentage of the total number of blows.

With the apparatus used, we were able to hit the target within an area of about $\frac{1}{8}$ in. in diameter. If a shallow V were used in the trough, the ball would wobble while rolling down the trough. The trough used had an included angle of 90 deg. which caused no difficulty.

MR. MARK B. MOORE.[13]—It seems to me that the comparative curves presented in Fig. 5 of the paper should tend to justify the use of repeated-bending fatigue data in cases where the actual loading is more of the repeated impact type. However, there still is a question as to the effect of speed on the results in the case of repeated impact.

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[11] William N. Findley, "The Fundamentals of Photoelastic Stress Analysis Applied to Dynamic Loading," *Proceedings*, 19th Semi-Annual Eastern Photoelasticity Conference, Cornell University, May, 1939.

[12] Research Fellow in Plastics, University of Delaware, Newark, Del.

[13] Assistant Professor of Mechanical Engineering, Pratt Institute, Brooklyn, N. Y.

One point which might be raised is this: The authors state that the impact stress can be considered as varying from zero to a maximum in one direction only. But, if the material is at all resilient, there will be a tendency for the specimen to "rebound" after the steel ball drops off. This might result in a case of reversed stress—not completely reversed, but certainly a stress range somewhat greater than just from zero to a maximum in one direction. Incidentally, an analysis of the behavior of the specimen under the impact of the steel ball *might* reveal that each impact is actually a succession of contacts between specimen and ball rather than one single impact.

MR. FINDLEY.—It is known that the free vibration of the specimen after the ball is thrown off results in a deflection of the specimen upward. Visual observation indicated that the magnitude of the upward deflection was small compared with the downward deflection. It should be possible to calculate the magnitude of the upward displacement, but the calculation would be quite involved. The displacement would probably depend upon the mass and modulus of elasticity of the specimen and of the ball, the geometry of the specimen and the internal friction (or damping) of the specimen. The authors hope to continue this study and to include a check on the calculated stresses (and deflections) by means of strain measurements.

The fact that a single impact is a succession of contacts under certain conditions has been shown both analytically and experimentally by certain investigators.[14,15] However, this condition is not likely to affect the calculations in the present instance since it is the maximum stress which causes the crack to start after repeated impact.

MR. STOCK.—I should like to ask Mr. Findley whether, in his fatigue work, he considers the possible errors that might be introduced by operating the flexural fatigue machine at higher than the fundamental vibration rate of the test specimens.

MR. FINDLEY.—That problem is, of course, of serious consideration in fatigue testing. In the machine which we used, the point of application of the exciting load was placed nearly at the center of percussion of the specimen and specimen holder (considered as hinged at the minimum section). Under this condition the moments tending to cause secondary bending of the system are small, so that the amplitude of such motions would be small except at resonance of the secondary modes of vibration. Even there the amplitudes of secondary modes of vibration would probably be small because of damping. Observation of the machine in motion indicated that the deflections in motion were substantially the same as those which occur at rest under the calculated load.

In a previously reported paper,[6] the fatigue machine was operated at different frequencies, varying from 40 cycles per minute to 3000 cycles per minute. The data obtained did not show any variation which could be attributed to secondary modes of vibration of the specimen.

MR. STOCK.—Mr. Findley's specimens were supported edgewise, so the fundamental frequency would have been considerably higher than that any way. 3000 cycles per minute would be 50 cycles per second; I doubt that one would come very close to the fundamental frequency of the specimen thus supported.

MR. FINDLEY.—If by "the fundamental frequency of the specimen thus supported" you mean the frequency of the specimen fixed in the grip and

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[14] S. Timoshenko, "Vibration Problems in Engineering," Second Edition, D. Van Nostrand Co., Inc., New York, N. Y., 1937, p. 394.

[15] H. L. Mason, "Impact on Beams," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 58, p. A55–A61 (1936).

restrained at the free end by the connecting rod, then your deduction is correct, as the frequency of this mode of vibration is large compared to the testing speed. This mode of vibration is referred to above as one of the secondary modes of vibration. The fundamental was considered to be the cantilever mode of vibration.

MR. J. O. ALMEN[16] (*by letter*).—The paper presented by Findley and Hintz is of unusual interest because, for the first time, we are given a continuous fatigue curve extending from one stress cycle to the endurance limit. A new and fruitful field for the study of strength of materials is opened to us.

The data suggest that by such tests as are reported we may eventually understand the significance of the impact test and that we may establish relationship between impact strength and fatigue strength. It has been evident for some time that fatigue data will plot as a straight line on log-log paper in the region between ten thousand and one million or more stress cycles. We now see that this straight-line relationship holds well up into the higher stress values in which the material tested is usually considered to be plastic as judged by static tests.

In log-log plotting we may, on the basis of this report, safely extrapolate much farther than has heretofore been permissible. If future tests confirm this finding there appears to be little need for conducting fatigue tests into large numbers of stress cycles.

It is hoped that Findley and Hintz will continue their tests on the same material using notched specimens to show (1) the relationship between impact and fatigue for such specimens, and (2) that a stress concentration factor can be expressed in terms of the slope of the fatigue curve. Another series of tests could profitably be made on specimens the tension loaded surfaces of which are compressively stressed by any convenient method to show their increased fatigue strength and the change in slope of the fatigue curve. The fatigue curves for notched, normal and pre-stressed specimens should converge toward a point in the vicinity of 12 stress cycles.

Messrs. Findley and Hintz have made a very good start on a most important phase of the fatigue problem. By their example perhaps other laboratories will be encouraged to conduct similar tests on other materials.

MR. FINDLEY.—Work is now in progress on repeated impact tests of notched specimens and it is hoped that results of tests such as suggested by Mr. Almen may be reported sometime in the future.

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FIELD AND LABORATORY DETERMINATION OF DISSOLVED OXYGEN[*]

By Robert C. Adams,[1] Robert E. Barnett,[2] and Daniel E. Keller, Jr.[3]

The Navy needs two methods for determination of dissolved oxygen: a simple field method for check on performance on shipboard of deaerating, feedwater heaters and an accurate laboratory method for evaluation of deaerators during contract-acceptance and type-approval tests. Evolution of a reliable laboratory method has involved some sixteen shipboard trials, eight type-approval tests of deaerating heaters of various sizes from different manufacturers, and extensive laboratory work over a period of about seven years. This paper gives details of laboratory and field methods and their apparatus and presents the results of tests by which the accuracy of these methods was evaluated.

Virtually all methods for the determination of dissolved oxygen in water are variants of that of Winkler in which the oxygen first oxidizes the manganous ion to manganic ion in alkaline solution followed by reaction of the manganic ion with iodide ion to yield free iodine stoichiometrically equivalent to the originally dissolved oxygen. The sample is then acidified to halt the preceding reactions and the free iodine titrated with standard sodium-thiosulfate solution. Lacking an entirely different method, the study described in this paper involved the trial and calibration of various modifications of apparatus and procedure for carrying out the Winkler method. The original plan, for calibration of the various methods which have been described in the literature and selection or adaptation of the most accurate, was never carried out. The immediate need for a method of ultimate accuracy for contract-acceptance tests of Naval deaerators precluded such a long investigation.

Both of the methods to be described have been calibrated and their errors have been either eliminated or evaluated. Study of previously published methods disclosed no procedure for calibration. While White, Leland and Button[4] and Schwartz[5] have demonstrated a high order of precision, and Schwartz and Gurney[6] recognized the interference of other substances with the determination of dissolved oxygen, no method has been reported for establishing the accuracy of an analytical determination of dissolved oxygen. The details of these two methods, the laboratory method first, are presented in the natural sequence: sampling, fixing and titration.

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[*] The opinions expressed in this paper are those of the writers and are not necessarily official opinions of the Engineering Experiment Station or the Navy Department.

Part of this paper was abstracted from a dissertation submitted to the faculty of the University of Maryland by Robert E. Barnett in partial fulfillment of the requirements for the degree of Master of Science.

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[3] Junior Chemist, U. S. Naval Engineering Experiment Station, Annapolis, Md.

[4] Alfred H. White, Claude H. Leland, and Dale W. Button, "Determination of Dissolved Oxygen in Boiler Feed Water," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 697 (1936).

[5] M. C. Schwartz, Louisiana State University Studies No. 21 (1935).

[6] M. C. Schwartz and W. B. Gurney, "The Determination of Traces of Dissolved Oxygen by the Winkler Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 796 (1934).

A sample to be satisfactory must be collected in a container which protects it from atmospheric contamination and which permits the addition of the fixing reagents without exposure to the air or entrapment of air bubbles. The McLean gas-sampling tube, modified as shown in Fig. 1, has been found best. Duplicate samples are necessary to correct for interferences, and two mated flasks having the same volume and the same stopcock-bore are connected in parallel using a Y outlet on the sample cooler. Since the stopcock-bore is about 2 mm. and the cooling coil usually has a diameter of 0.25-in., the two stopcocks in each flask provide an orifice restriction which equalizes the rate of flow through the two flasks. This equality of flow should be assured by mounting the flasks at the same level and either omitting drain tubing from the upper ends or providing equal siphon discharge by having the outlet ends of the drain tubing at the same level.

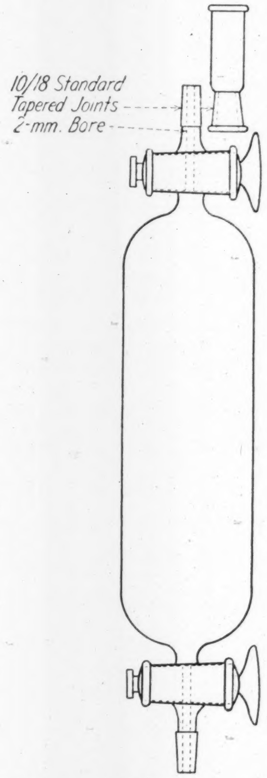

FIG. 1.—500-ml. Dissolved Oxygen Sampling Tube.

Cooling of samples more than 10 F. below room temperature is unnecessary. The sample should be cooler than room temperature so that the effect of heat transfer to the sample will be to increase the pressure within the flask. The cooler recommended is the double-coil, counter-current type illustrated in Fig. 2. The sample flow should be controlled at the outlet of the cooler while the cooling-water flow is controlled at its inlet to the cooler. The tightness of both coils of the cooler should be assured by hydrostatic test.

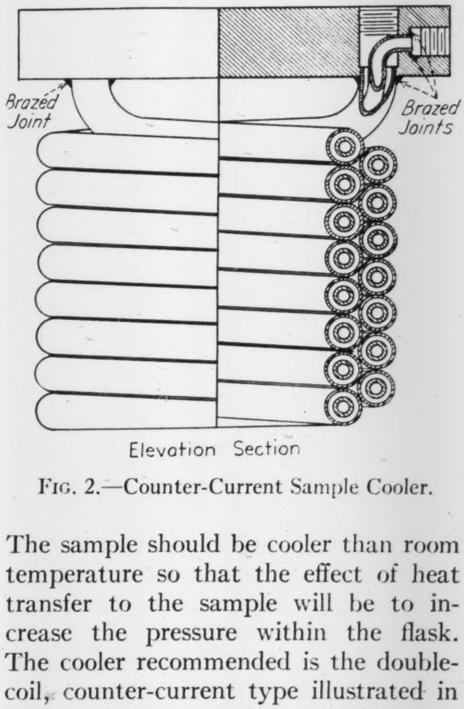

FIG. 2.—Counter-Current Sample Cooler.

The rising water surface dissolves atmospheric oxygen when the flasks first are connected to the sampling line.

Sampling flow must be continued until all of this contaminated water has been flushed out and the sample in the flask is representative. The minimum number of changes required is about seven, as illustrated in Fig. 3, so that at least ten are recommended. This is based on a rate of flow sufficient to fill the sampling flask in 40 to 60 sec. At lower rates of flow more changes should be provided. When the flushing is completed, stopcocks on both flasks are closed simultaneously by pairs.

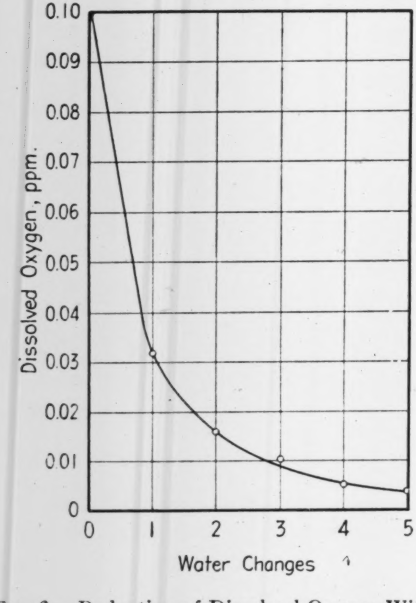

Fig. 3.—Reduction of Dissolved Oxygen With Changes in Sample.

With two identical samples collected, the operator proceeds with the fixing. At this point one of the samples must be designated as the *sample* and the other as the *blank*. Directions for preparation of the reagents are given in the Appendix. The fixing reagents differ from those usually employed in one important respect. The alkaline-iodide solution contains free iodine in an amount sufficient to combine with the reducing impurities in the sampled water. Since high concentrations of iodine unavoidably reduce the accuracy of the determination, the minimum necessary amount should be used. It is convenient to prepare several alkaline-iodide solutions containing varying concentrations of iodine.

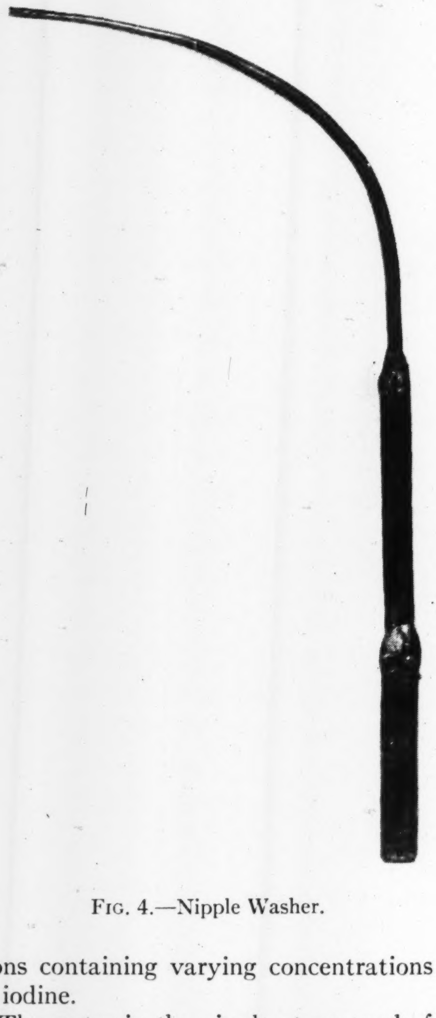

Fig. 4.—Nipple Washer.

The water in the nipple at one end of the *sample* flask is flicked out and reagent cup No. 1 is affixed. Two (2.00) milliliters, plus the predetermined capacity of the stopcock-bore, of iodized-alkaline-iodide solution is added to the reagent cup. The error in measurement of the fixing reagents must not exceed 0.02 ml. The plug of the upper stopcock is rotated 180 deg. to make sure that no air is trapped in the bore and the upper nipple is inspected closely for any air bubble trapped below the reagent. If air is present it can be released by probing with a clean copper wire. The lower stopcock, that stopcock being closed when the meniscus just reaches the surface of the plug, and the lower stopcock closed immediately afterward. Reagent cup No. 1, which is used only for the iodized-alkaline-iodide solution, is removed, rinsed thoroughly with distilled water, and laid aside in a clean place to drain completely.

Both nipples are rinsed thoroughly, either with a wash bottle or with a tubing device such as shown in Fig. 4. Water is flicked out of the nipple opposite that used for the first addition and a second reagent cup is affixed. Two milliliters of manganous sulfate solution are added to the cup, then added to the sample by the previous procedure. The reagent cup is removed and rinsed. The sample is thoroughly mixed and then laid aside while 2-ml. additions of iodized-alkaline-iodide, sulfuric acid and manganous sulfate are made, in that order, to the *blank*. Finally, a 2-ml. addition of acid is made to the *sample*. The same precautions are observed and procedure followed in all additions. Either of the other reagents may be added through the same nipple as the acid, but iodized-alkaline-iodide and manganous-sulfate solutions must be added through *opposite* ends of the sampling flask to avoid fouling of nipple and stopcock passages with manganous hydroxide.

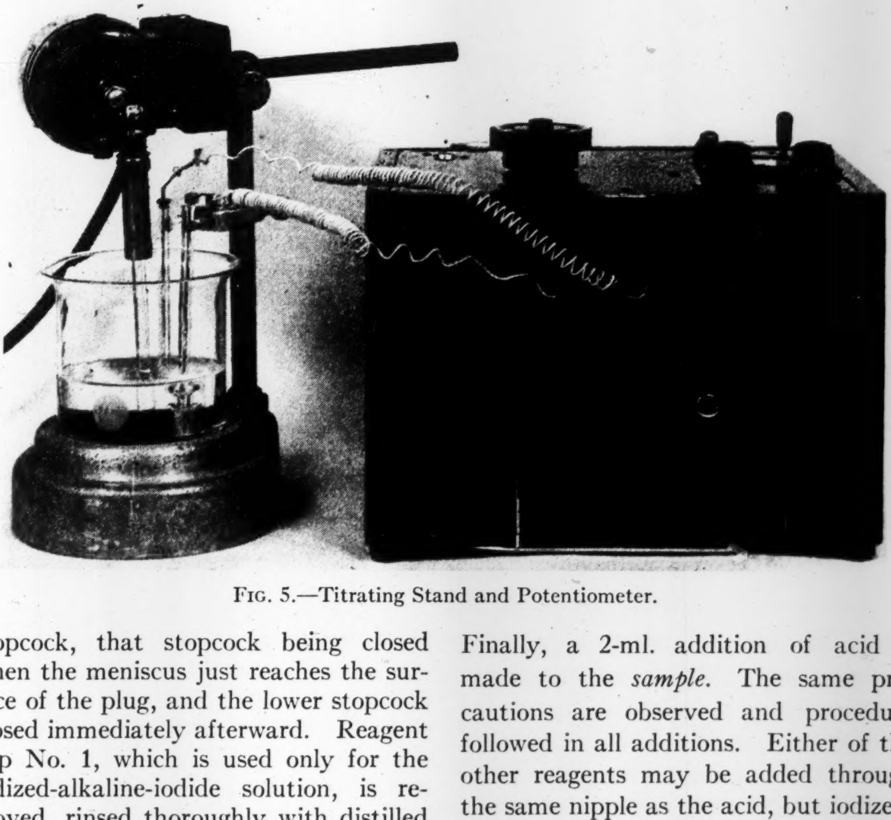

FIG. 5.—Titrating Stand and Potentiometer.

The fixed samples can be exposed to the atmosphere for titration, but titration should be completed within 30 min. It is preferable to titrate immediately after fixing.

The apparatus for titration, shown in Fig. 5, comprises a titrating stand and a potentiometer. The titrating stand consists of two parts: a cast-aluminum base with an opening to fit an 800-ml. Griffin beaker; and a separable support rod, squared at the end to fit a hole in the base, which carries an adjustable support for the electrodes and, at the upper end, a clamp for the electric stirrer. With this arrangement, the sample for titration is first emptied from the McLean flask into an 800-ml. beaker, then the electrode and stirrer assembly is lifted free while the sample beaker is put in place, and the assembly replaced. The stirrer should be of variable-speed type with the variable-speed adjustment securely mounted so that it can be operated with one hand during a titration. The Beckman pH meter was chosen as the potentiometer because it can be operated with one hand. Saturated-calomel and platinum electrodes are used.[7]

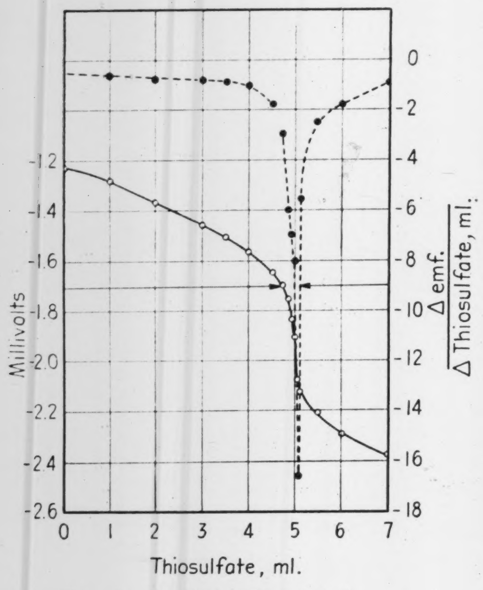

FIG. 6.—Specimen Titration Curves.

At the start of a titration the analyst fills a 1.00-ml. or 5.00-ml. graduated pipette, held in the left hand, with standard sodium thiosulfate solution, adjusts the speed of stirring of the sample, takes an initial reading of the potential across the electrodes and records it. The thiosulfate is added in increments, a potential reading being made and recorded after each. The additions of thiosulfate are reduced progressively to the smallest amount which can be measured with the pipette as the end point is approached. Two or three additions beyond the end point are sufficient for its identification.

The end point is the minimum on the first differential of the thiosulfate-emf. curve. The thiosulfate-emf. curve and its first differential for a specimen titration are shown in Fig. 6. With a sufficiently accurate potentiometer the end point can be selected from the titration record without plotting of the curve. As the titration proceeds, and each successive potentiometer reading is recorded, the division: $\frac{\Delta \text{ emf.}}{\Delta \text{ thiosulfate}}$ is recorded in a third column. The minimum value of this quotient corresponds to the end point. If two successive quotients have essentially the same minimum value the amount of thiosulfate halfway between then is taken as the end point of the titration, but no more refined interpolation is justified.

After both the sample and the blank

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[7] G. A. Perley, "Determination of Dissolved Oxygen in Aqueous Solutions," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 11, May 15, 1939, p. 240.

have been titrated, the amount of dissolved oxygen in the water sampled must be calculated from these values. The amount of thiosulfate required in either case corresponds to the algebraic sum of four components:

1. The oxygen dissolved in the water sampled;
2. The oxidizing or reducing impurities in the water sampled;
3. The dissolved oxygen introduced with the fixing reagents;

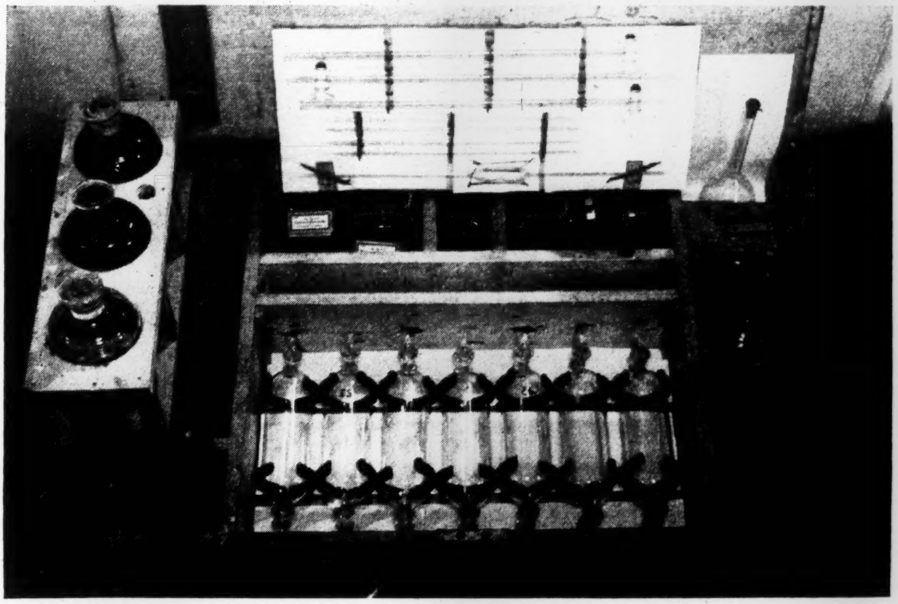

FIG. 7.—Shipping Chest for Laboratory Method.

4. The iodine introduced with the iodized-alkaline-iodide solution.

Items 1 and 2 are proportional to the size of the sample and are measured as parts per million of dissolved oxygen; items 3 and 4 are fixed by the analyst, do not vary with sample volume, and should be measured as milliliters of equivalent sodium thiosulfate. Since no oxygen is fixed in the blank the titration of the blank amounts to the sum of items 2 and 4 only.

Accurate calculation of the amount of dissolved oxygen in the sampled water consists of five steps:

A. Subtract from the titration of the sample the predetermined equivalent milliliters of thiosulfate for the sum of items 3 and 4;

B. Multiply the difference obtained in A, above, by the factor:

$$\frac{\text{normality of thiosulfate} \times 8000}{\text{volume of sample (ml.)}}$$

C. Subtract from the titration of the blank the thiosulfate equivalent of item 4;

D. Multiply the difference from C by the corresponding factor for the flask used in collection of the blank;

E. The value from B minus that from D equals ppm. of dissolved oxygen in the sample.

Usually the calculation can be abbreviated with considerable saving in time and no loss in precision. If all sampling flasks have capacities within 2 per cent of the average, the capacities of the two flasks in each pair differ by no more than 1 per cent, and the sum of items 1 and 2 (without regard to sign) does not exceed 0.10 ppm., a simpler calculation is possible. In such cases the difference in titrations of sample and blank is multiplied by the mean factor for the two flasks and item 3, calculated in ppm. for the mean capacity of all the flasks, is subtracted from the product.

The method described above was developed primarily for the highest possible precision, for use as a laboratory or referee method. When it has been necessary to extend this precision to determinations made in manufacturing plants or on shipboard, a shipping chest is used as shown in Fig. 7. Sample coolers usually must be installed in advance and the potentiometer preferably is carried by the analyst. Distilled water is the only material which must be provided at the point of use.

The high order of accuracy required for research and qualification tests is unnecessary for routine control tests. In the latter case it is necessary only to detect malfunctioning of deaerating equipment before serious corrosion of economizer tubing can occur. Apparatus and procedure for this less exacting purpose can be simplified with reduction in the cost of the necessary equipment. The ultimate in simplification is necessary because of the desirability of making frequent determinations where the only available analysts are without laboratory training or experience.

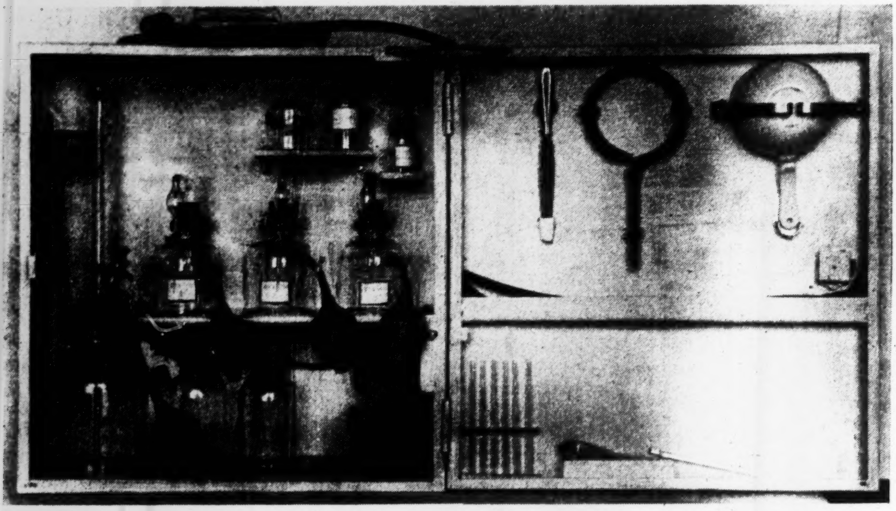

FIG. 8.—Navy Dissolved-Oxygen Testing Cabinet.

The laboratory method has been described. Figure 8 shows the dissolved-oxygen testing cabinet in use on Naval vessels[8] or for field analysis. The calibration of both methods is discussed in the last section of the paper.

In field work, samples are collected in 300-ml. B.O.D. bottles which have a raised lip at the neck and a glass stopper with a conical point. The capacity of such bottles can be maintained at 300 ± 5 ml. in manufacture. A cooler, as shown in Fig. 2, should be used, with its outlet connected with rubber tubing to a short length of clean glass tubing which extends to the bottom of the sample bottle. The sample flow should be continued and permitted to overflow to waste until the contents of the bottle have been changed about ten times.

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[8] Navy Department, Bureau of Ships Ad Interim Specification 57T6 (INT), April 15, 1940.

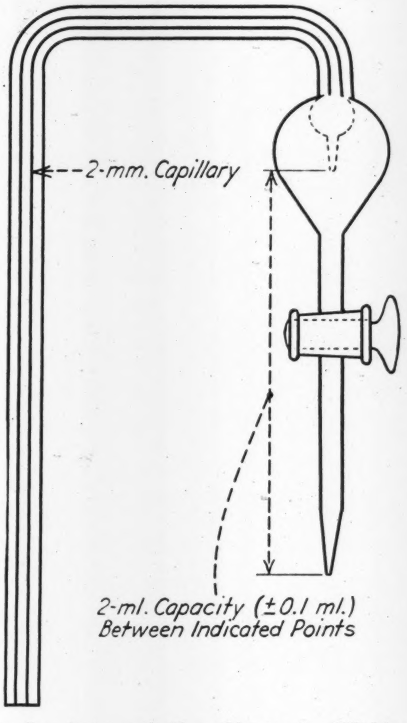

FIG. 9.—Automatic-Zero Pipette for Winkler Reagents.

The glass delivery tube is withdrawn carefully to avoid disturbing the surface of the water, and the wetted stopper is immediately floated down into place and then twisted to seat it securely. The stoppered bottle should be inverted, inspected, and the sample should be discarded if any bubbles of air are detected.

Since starch is used as indicator in the subsequent titration, the temperature of the collected sample must not exceed 70 F. After-cooling of the collected sample by immersion in ice water or by a stream of ice water is undesirable because the reduction of pressure within the bottle may be sufficient to draw in contaminated water or air.

The Winkler fixing reagents are added by means of the pipettes shown in Fig. 8 and detailed in Fig. 9. These pipettes are drained when not in use so that all three should be filled by air pressure in the reagent bottles. Enough reagent is then drained from each to fill the capillary tip below the stopcock, and the pipettes are refilled. The stopper is removed from the sample bottle, which is raised onto the tip of the manganous-sulfate pipette as the stopcock of the pipette is opened. As the meniscus in the pipette drops below the stopcock the sample bottle is lowered and, without replacing the stopper, is raised immediately onto the tip of the alkaline-iodide pipette where the second addition is made in the same fashion. The stopper is replaced, the bottle inverted or swirled to mix thoroughly the sample and reagents, and then allowed to stand while the precipitate settles. As soon as the precipitate has settled below the shoulder of the sample bottle, the acid addition should be made from the third pipette in the fashion described above. The bottle stopper is replaced and the contents mixed thoroughly. As soon as all precipitate has been completely dissolved, the sample is fixed and ready for titration.

Fresh starch solution should be prepared for use in the titration. A hot plate and a beaker are provided in the cabinet, Fig. 8, so that 100 ml. of distilled water can be rapidly heated to the boiling point. About 1 g. of chemi-

TABLE I.—REACTIONS IN WINKLER METHOD, BINIODATE MODIFICATION.

	Addition Numbers Reagents Added	Constituents Present in Samples Prior to Addition of Reagent	Normal Winkler Reaction And Possible Redox Reaction	Constituents Present In Samples After Addition of Reagent	Remarks
SAMPLE	1. Manganous sulfate, $MnSO_4$	Dissolved O_2 Unknown Oxidizing and Reducing (Redox) ions.	None (*a*) $Mn^{++} + Redox \rightarrow Mn^{++++}$	Mn^{++}; O_2 Mn^{++++}(?) Redox ions	Solution is neutral. (*a*) Any Redox impurity having this effect would be indistinguishable from oxygen.
	2. Alkaline potassium iodide, KOH—KI	Mn^{++} Mn^{++++} Redox O_2	$MnSO_4 + 2KOH \rightarrow Mn(OH)_2 + K_2SO_4$ $2Mn(OH)_2 + O \rightarrow 2MnO(OH)_2$ $MnO(OH)_2 + KI + H_2O \rightarrow Mn(OH)_2 + I_2 + 2KOH$ (*b*) $Mn^{++++} + Redox \rightarrow Mn^{++}$ (*c*) $KI + Redox \rightarrow I_2$ (*d*) $I_2 + Redox \rightarrow I^-$	Mn^{++} I^- I_2 Redox OH^-	Solution is alkaline. (*b*) If such a reaction as this occurs only in alkaline solution, it would not occur in the blank and error would result. However, the coexistence in the same sample with O_2 of any such substance is highly improbable. (*c*) This reaction is compensated for by a similar reaction in the blank, since the KI is present there in alkaline solution also. (*d*) This reaction is not compensated for by any similar reaction in the blank since no free I_2 is present at this stage.
	3. Sulfuric acid, H_2SO_4	Mn^{++} I^- I_2 Redox OH^-	$2H_2SO_4 + Mn(OH)_2 + 2KOH \rightarrow K_2SO_4 + MnSO_4 + H_2O$ (*e*) $Mn^{++} + Redox \rightarrow Mn^{++++}$	I^- Mn^{++} H^+ I_2 Mn^{++++}(?) Redox	Solution is acid. Redox reactions (*a*), (*b*), (*c*), (*d*), (*e*) are all possible at this stage of the process; however, if any of these take place, they could be compensated for by similar reactions in the blank.
	4. Potassium biniodate, $KH(IO_3)_2$	Mn^{++} Redox H^+ I^- I_2 Mn^{++++}(?)	$KH(IO_3)_2 + 10KI + 11H_2SO_4 \rightarrow 6I_2 + 11KHSO_4 + 6H_2O$	Same as in Step 3 above.	Solution is acid. Same as Step 3.
	5. Titration sodium thiosulfate, $Na_2S_2O_3$	Same as Step 4 above.	$2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$	Mn^{++} H^+ I^- Redox $Na_2S_4O_6$	Solution is acid. All reactions including any Redox reactions and the normal iodine titration with thiosulfate taking place in this stage are compensated for by corresponding reactions in the blank.
BLANK	1. Alkaline potassium iodide, KOH—KI	Dissolved O_2, Unknown oxidized and reducing (Redox) ions.	None	O_2 Redox OH^- I^-	Solution is alkaline. A reaction similar to reaction (*c*) is possible but would be compensated for. Since no *iodine* is present at this stage, the solution being alkaline, reaction (*d*) is not compensated for.
	2. Sulfuric acid, H_2SO_4	O_2 Redox OH^- I^-	$H_2SO_4 + 2KOH \rightarrow K_2SO_4 + 2H_2O$	O_2 Redox H^+ I^-	Solution is acid. The only Redox reaction which could take place would be compensated for as it is similar to reaction (*c*) in acid solution. Reaction (*d*) cannot be compensated for at this stage as no I_2 is present.
	3. Manganous sulfate, $MnSO_4$	O_2 Redox H^+ I^-	None (*f*) $Mn^{++} + Redox \rightarrow Mn^{++++}$(?)	Mn^{++} O_2 Redox H^+ I^-, Mn^{++++}(?)	Solution is acid. Reactions similar to (*a*) and (*c*) are possible but would be compensated for.
	4. Potassium biniodate, $KH(IO_3)_2$	Mn^{++} O_2 Redox H^+ I^- Mn^{++++}(?)	$KH(IO_3)_2 + 10KI + 11H_2SO_4 \rightarrow 6I_2 + 11KHSO_4 + 6H_2O$	Mn^{++} H^+ I^-, I_2 O_2 Redox Mn^{++++}(?)	Solution is acid. Same as above except that reaction (*d*) is now possible since I_2 is now present.
	5. Titration sodium thiosulfate, $Na_2S_2O_3$	Mn^{++} O_2 Redox H^+ I^- I_2 Mn^{++++}(?)	$2Na_2S_2O_3 + I_2 - Na_2S_4O_6 + 2NaI$	Mn^{++} H^+ I^- O_2 Redox $Na_2S_4O_6$	Solution is acid. Same as Step 5 in oxygen sample. The presence of O_2 here has no effect upon the result.

TABLE II.—REACTIONS IN WINKLER METHOD, NAVY LABORATORY MODIFICATION.

	Addition Numbers Reagents Added	Constituents Present in Samples Prior to Addition of Reagent	Normal Winkler Reaction And Possible Redox Reaction	Constituents Present In Samples After Addition of Reagent	Remarks
SAMPLE	1. Alkaline potassium iodide + iodine, $KOH—KI—I_2$	Dissolved O_2, unknown oxidizing and reducing (Redox) ions.	None (*a*) $I_2 + \text{Redox} \rightarrow I$ (*b*) $I^- + \text{Redox} \rightarrow I_2$	OH^- I^- I_2,O_2 Redox	Solution is alkaline. (Iodine is added in measured amount to the sample with the reagent. It is present in the reagent as potassium hypoiodite KOI). (*a* and *b*) Reactions (*a*) and (*b*) are compensated for by similar reactions in the blank.
	2. Manganous sulfate, $MnSO_4$	OH^- I^- I_2 Redox O_2	$MnSO_4 + 2KOH \rightarrow Mn(OH)_2 + K_2SO_4$ $2Mn(OH)_2 + O_2 \rightarrow 2MnO(OH)_2$ $MnO(OH)_2 + KI + H_2O \rightarrow Mn(OH)_2 + I_2 + 2KOH$ (*c*) $Mn^{++} + \text{Redox} \rightarrow Mn^{++++}$ (*d*) $Mn^{++++} + \text{Redox} \rightarrow Mn^{++}$	Mn^{++} OH^- I_2 from reagent I_2 from oxygen Redox I^-	Solution is alkaline. (*c*) Any Redox impurity having the effect of reaction (*c*) would be indistinguishable from oxygen by this method. (*d*) The presence of a redox impurity having the effect of reaction (*d*) would result in an error which is not compensated for by any corresponding error in the blank. However, the co-existence of any such substance with oxygen is improbable since it presumably should react with oxygen.
	3. Sulfuric acid, H_2SO_4	Mn^{++} OH^- I_2 from reagent I_2 from oxygen Redox I^-	$2H_2SO_4 + Mn(OH)_2 + 2KOH \rightarrow K_2SO_4 + MnSO_4 + 4H_2O$ (*e*) $Mn^{++} + \text{Redox} \rightarrow Mn^{++++}$	Mn^{++} H^+ I_2 from reagent I_2 from oxygen Redox I^- Mn^{++++}(?)	Solution is acid. Any Redox reaction taking place at this stage is compensated for by similar reactions taking place in the blank.
	4. Titration sodium thiosulfate, $Na_2S_2O_3$	Mn^{++} H^+ I_2 from reagent I_2 from oxygen Redox I^- Mn^{++++}(?)	$2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$	Mn^{++} H^+ Redox I^- $Na_2S_4O_6$	Solution is acid. Same as Step 3.
BLANK	1. Alkaline potassium iodide, $KOH—KI—I_2$	Dissolved O_2, unknown oxidizing and reducing (Redox) ions.	None (*f*) $I_2 + \text{Redox} \rightarrow I^-$ (*g*) $I^- + \text{Redox} \rightarrow I_2$	OH^- O_2 I^- Redox I_2 from reagent I_2 from Redox (?)	Solution is alkaline. Same as Step 1 in the sample. Reaction (*f*) and (*g*) cancel similar reactions (*a* and *b*) in the sample.
	2. Sulfuric acid, H_2SO_4	OH^- Redox O_2 I^- I_2 from reagent I_2 from Redox (?)	$H_2SO_4 + 2KOH \rightarrow K_2SO_4 + H_2O$	H^+ O_2 I^- I_2 from reagent Redox	Solution is acid. Same as Step 3 in the sample except for reaction (*e*).
	3. Manganous sulfate $MnSO_4$	H^+ O_2 I^- I_2 Redox	None (*h*) $Mn^{++} + \text{Redox} \rightarrow Mn^{++++}$	Mn^{++} Mn^{++++}(?) H^+ O_2 Redox I_2, I^-	Solution is acid. Same as Step 3 in the sample. Reaction (*e*) is here compensated for by reaction (*h*).
	4. Titration sodium thiosultate, $Na_2S_2O_3$	Mn^{++} Mn^{++++} (?) H^+, O_2 I^- I_2 Redox	$2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$	Mn^{++} H^+ O_2, I^- $Na_2S_4O_6$ Redox	Solution is acid. Same as Step 4 in the sample.

cally pure soluble starch is sprinkled into the boiling water from the tip of a spatula. The solution is stirred briefly and permitted to boil for a half minute before the beaker is removed from the hot plate. As soon as this solution is cool it is ready for use, a portion being transferred to the dropping bottle for the purpose.

The sample is titrated with 0.0075 *N* sodium thiosulfate solution. This is made by dilution of a 0.15 *N* stock

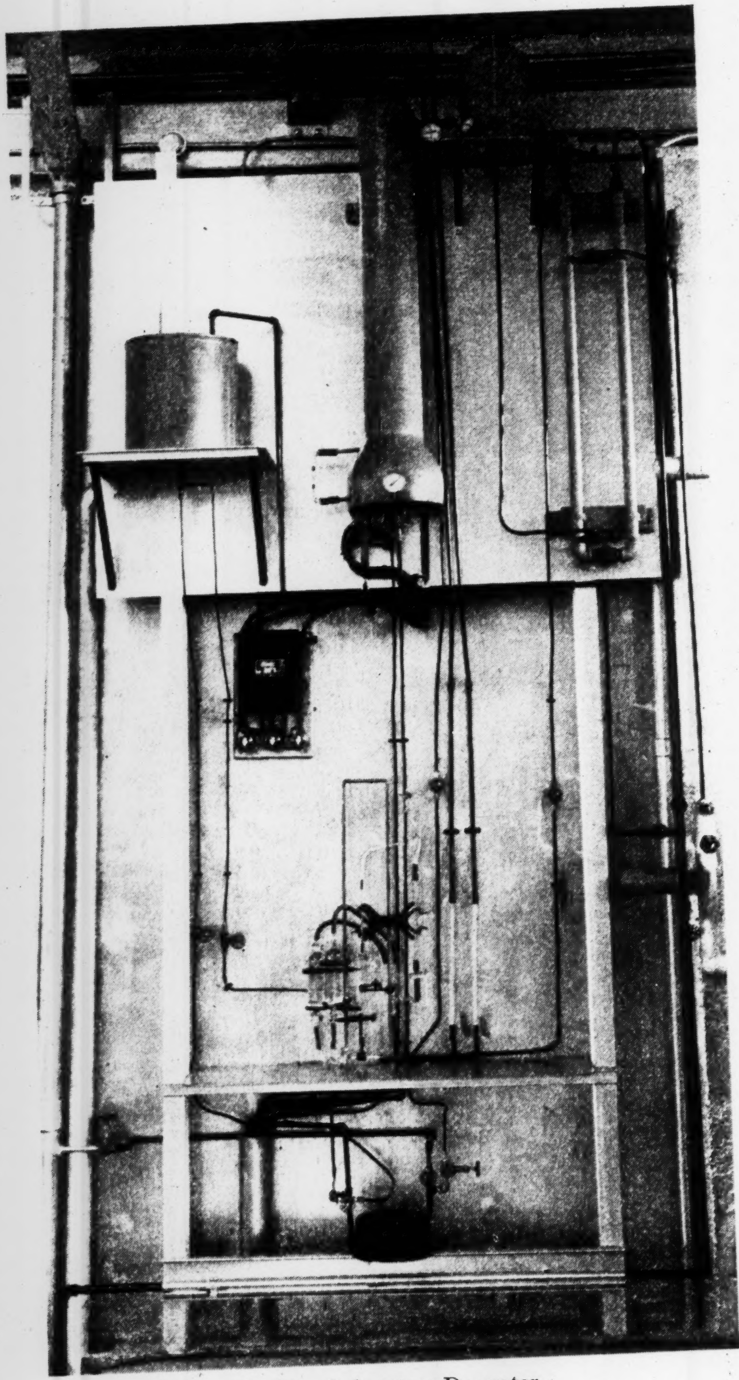

FIG. 10.—Laboratory Deaerator.

solution which is stabilized with potassium furoate[9] so that it can be stored for long periods. The diluted reagent solution contains insufficient inhibitor for stabilization and must be discarded after seven days.

For titration, the entire contents of the sample bottle are emptied into the clean, porcelain casserole; the automatic-zero burette is filled with thiosulfate; and several drops of starch indicator are added to the sample. Titration proceeds in the usual manner until the blue, starch-iodide color is just destroyed. The burette reading at this point, when multiplied by 0.2 equals the dissolved-oxygen content of the sample in parts per million.

The laboratory and field methods which have been described were developed in the search for methods of known accuracy which could be applied with assurance to their separate purposes. Calibration and simultaneous improvement of the laboratory method involved investigation of its inherent errors as well as development of a reproducible analytical procedure. The most helpful tool in the search and elimination of potential and actual errors was the tabulation of the net results of all possible reactions of each step in the procedure. Table I disclosed a possibility of error in the biniodate modification of the Winkler method which has not been reported. At one stage in fixing the sample, free iodine is present in alkaline solution while this does not occur in the blank. Thus if an impurity is present which reduces iodine in alkaline solution, but not in acid solution, an erroneously low value for dissolved oxygen results. Systematic test by introduction of various ions into water of known dissolved-oxygen content disclosed that ferrous ion reacts with iodine in this manner.

In order to eliminate the error from ferrous ion and similarly reacting redox impurities it was necessary to provide free iodine in alkaline solution in both sample and blank. Table II details the net results of both normal and side reactions in this procedure and shows there are no unmeasured side reactions.

For the purpose of calibration, a laboratory deaerator was designed and constructed. This is illustrated in Fig. 10. It consists of three parts: a heater, a preboiler, and a stripping column. The heater consists of a U-shaped, two-pass, tubular heat exchanger, the steam jackets of which are connected in parallel. The feedwater is brought to the atmospheric boiling point in its passage through the heater, then goes to the preboiler. The preboiler is a cylindrical kettle with three vertical baffles which force the water to flow in a serpentine, horizontal path from one side to the other. It has a false bottom extending to within ½ in. of the walls, below which wet steam and condensate from the heater jacket are admitted. The rate of admission is controlled to maintain continuous, violent boiling at this stage. The boiling water enters the top of the insulated column where it is distributed over the column packing of ¼-in. Pyrex Raschig rings. Electric heaters on the bottom of the column pot maintain a continuous and copious flow of steam upward through the falling water, stripping the dissolved gases and discharging them from the column vent. This empirical apparatus is not efficient and not completely effective in removal of dissolved gases, but it has the merit of delivering water of low and constant dissolved-oxygen content.

For the field method, the oxygen-free effluent from the deaerator was mixed with controlled small proportions of water containing a determined, large amount of dissolved oxygen. Two small rotameters were installed for the requisite measurement of flows for accurate proportioning. The larger, with a maximum capacity of 1040 ml. per min., was installed in the discharge from the cooler on the deaerator sampling line; the smaller, having a range of 0 to 70 ml. per min., on the discharge from an overhead storage tank for saturated water. Connections were provided for simultaneous sampling of both waters and their mixture. Table III presents the data from calibration of the field method. These indicate that both the sensitivity and the accuracy of this method are of the order of 0.02 ppm.

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[9] A. M. Platow, *The Chemist-Analyst*, Vol. 28, p. 30 (1939).

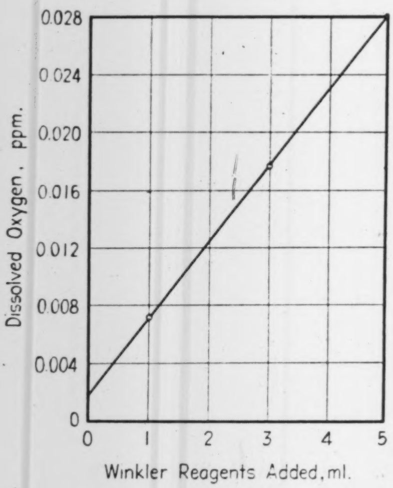

FIG. 11.—Dissolved Oxygen in Winkler Reagents.

The dissolved oxygen introduced to the sample in the fixing reagents was determined by use of the laboratory deaerator. A series of samples of deaerated water was fixed with 1.0, 3.0, and 5.0 ml. of each of the reagents and the fixed samples titrated. The straight line of Fig. 11 was obtained by plotting the oxygen equivalent of the thiosulfate requirement *versus* the milliliters of each reagent. The slope of this line equals 0.052 ppm. per milliter of reagents, indicating that the correction for dissolved oxygen introduced with the fixing reagents, when 2 ml. of each is used, is 0.0104 ppm.

The most convincing evidence of the precision and accuracy of the laboratory method was collected during a shop test of a Naval deaerating feed tank. Table IV gives complete calculations for six successive determinations of the dissolved-oxygen content of the effluent from this heater. Although there was a slight change in rate of flow between samples Nos. 3 and 4, it will be noted that the range from maximum to minimum value is only 0.0020 ppm. and the mean of all determinations was less than

TABLE III.—CALIBRATION DATA FOR FIELD METHOD.

Deaerated Water Flow, ml. per min.	Saturated Water Flow, ml. per min.	Calculated O_2 Content, ppm.	Determined O_2 Content, ppm.	Error
580	76	0.910	0.914	+0.004
580	58	0.714	0.743	+0.029
580	46	0.584	0.580	−0.004
580	30.5	0.393	0.403	+0.010
580	19.0	0.250	0.251	+0.001
580	9.5	0.127	0.146	+0.019
580	3.5	0.047	0.079	+0.032
580	0.75	0.010	0.040	+0.030

TABLE IV.—RESULTS FROM SUCCESSIVE DETERMINATIONS BY THE LABORATORY METHOD.

		Time	Thiosulfate, ml.	O_2 in Sample, ppm.	O_2 in Blank, ppm.	Difference in Sample—Blank	Final O_2, ppm. Corrected by Subtracting 0.0104 ppm.
No. 1	Sample	1046	0.24	0.0187		0.0113	0.0009
	Blank	1046	0.095	...	0.0074	...	
No. 2	Sample	1117	0.29	0.0224		0.0117	0.0013
	Blank	1117	0.14	...	0.0107		
No. 3	Sample	1128	0.34	0.0265		0.0109	0.0005
	Blank	1128	0.20	...	0.0158		
No. 4	Sample	1139	0.36	0.0275		0.0113	0.0009
	Blank	1139	0.21	...	0.0162		
No. 5	Sample	1212	0.305	0.0238		0.0097	−0.0007
	Blank	1212	0.18	...	0.0141		
No. 6	Sample	1223	0.225	0.0174		0.0098	−0.0006
	Blank	1223	0.10	...	0.0076		

0.0004 ppm., which may be considered essentially zero.

From these data it may be concluded that the laboratory method which has been described will yield values of dissolved oxygen with a precision of 0.001 ppm. and within 0.002 ppm. of the true values. The field method will detect as little as 0.02 ppm. of dissolved oxygen and measure the concentration with an accuracy of about 0.02 ppm. Both methods have the order of accuracy for which they were intended, combined with the greatest simplicity which several years of study and trial have accomplished.

Acknowledgment:

The authors have been assisted by the suggestions and criticisms of so many persons interested in the determination of dissolved oxygen that detailed acknowledgments are impossible. They wish to express their particular appreciation to Captain T. J. Bay, U.S.N., for his confidence in the accuracy of their reported results.

APPENDIX

PREPARATION OF REAGENTS

A. *Laboratory Method:*

1. *Iodized Alkaline Iodide.*—Dissolve 700 g. of KOH in 700 ml. of distilled water and cool to room temperature. Dissolve 150 g. of KI in 200 ml. of distilled water, cool to room temperature, and mix with the KOH solution in a 1000-ml. volumetric flask. Wash approximately 0.03 g. of crystalline I_2 (proportionately more if high interference is encountered) into the flask, dissolve, and dilute to 1000 ml. with distilled water. Store in a brown bottle.

2. *Manganous Sulfate.*—Weigh 480 g. of $MnSO_4 4H_2O$ into a 600-ml. beaker. Add 200 ml. of distilled water, stir well, and transfer the supernatant solution to a 1000-ml. volumetric flask. Repeat three times, wash the last of the crystals into the flask, shake the flask until solution is complete, and dilute to 1000-ml. with distilled water.

3. *Sulfuric Acid.*—Pour carefully 750 ml. of concentrated H_2SO_4 (sp. gr. 1.84) into 250 ml. of distilled water in a 1000-ml. beaker. Cool to room temperature and transfer to a 1000-ml. volumetric flask. Dilute to 1000-ml. with distilled water.

4. *Stock Sodium Thiosulfate, 0.05 N.*—Dissolve 12.42 g. of $Na_2S_2O_3 \cdot 5H_2O$ in distilled water. Dilute to approximately 800 ml. in a 1000-ml. volumetric flask. Add 10 g. of potassium furoate, $C_4H_3O \cdot CO_2K$, shake thoroughly to dissolve and dilute to 1000 ml. with distilled water. Standardize against $K_2Cr_2O_7$ or KH $(IO_3)_2$ by electrometric titration.

5. *Reagent Sodium Thiosulfate, 0.005 N.*—With a calibrated pipette transfer 25.00 ml. of stock solution to a 250-ml. volumetric flask. Dilute to the mark with distilled water and mix completely.

6. *Preparation of Potassium Furoate.*—Dissolve pure, white, crystalline furoic acid, Eastman No. 1366, in ethanol. Neutralize with a solution of KOH in ethanol to pH 7.0. Evaporate to dryness on a steam bath.

B. *Field Method:*[10]

1. *Manganous Sulfate.*—Same as for laboratory method.

2. *Alkaline Iodide.*—Same as for laboratory method except that no iodine is added.

3. *Sulfuric Acid.*—Same as for laboratory method.

4. *Stock Sodium Thiosulfate, 0.15 N.*—Dissolve 37.23 g. $Na_2S_2O_3 \cdot 5H_2O$ and 10 g. potassium furoate in one liter of solution following the same procedure as for the laboratory method.

5. *Reagent Sodium Thiosulfate, 0.0075 N.*—Rinse the reagent bottle, Fig. 8, and a 50-ml. pipette with distilled water. Rinse and then fill the pipette with a stock sodium-thiosulfate solution. Drain the pipette into the reagent bottle. Fill the bottle to the 1000-ml. mark with distilled water, stopper, shake thoroughly, and replace the stopper with that containing the burette. Fill the burette and drain to waste. Discard the remainder of this solution after 1 week.

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[10] Navy Department, Bureau of Ships Ad Interim Specification 51C29 (INT), August 15, 1941.

DISCUSSION

Mr. J. F. Sebald[1] (*presented in written form*).—The availability of an accurate and reliable method for the determination of dissolved oxygen is of primary importance to the manufacturer of deaerating equipment such as surface condensers and pressure deaerators. The degree of design perfection of such apparatus is entirely dependent upon the accuracy and reliability of the test method employed to measure its performance. Because the reputation and financial investment of the manufacturer of deaerating equipment is largely dependent upon reliable and accurate test methods the question of accuracy is probably of greater importance to us than to a referee laboratory.

We feel that the test method developed by Messrs. Adams, Barnett, and Keller is a great step forward in the field of dissolved oxygen testing.

Mr. Adams has mentioned that this method was evolved, in part, during tests made on deaerating equipment for the Naval program. Since the company with which I am associated was one of those manufacturing some of this equipment I feel that we have had considerable experience with this method as we have seen it applied by the Engineering Experiment Station and have experimented with it ourselves. There appears to be certain advantages in the Naval Laboratory method over both the biniodate modification of the Winkler method and the Schwartz and Gurney type B method and an evaluation of the three with waters containing various types of interference should be a great contribution to the art.

Our research testing for the purpose of comparing the biniodate modification and the Navy Laboratory modification of the Winkler test indicates a chemical superiority of this latter method over the biniodate modification as previously used by the Engineering Experiment Station and by our research department.

This chemical superiority of the method lies in part to the reduction of error due to an arbitrary reagent correction necessary in the biniodate procedure in order to avoid disproportionate negative results from oxygen-free samples.

Such an arbitrary or modified reagent correction results also in disproportionately high readings from oxygen bearing samples. The magnitude of such an error could easily penalize the manufacturers of deaerators as much as 30 per cent of the allowable oxygen limit.

The Navy Laboratory modification of the Winkler test permits the use of a reagent correction of the magnitude reported by Messrs. White, Leland, and Button in 1936,[2] without employing any arbitrary corrections.

I should like to point out that the accuracies reported by Mr. Adams in Table IV represent a test of less than three hours duration. We have found similar accuracies are readily obtainable over such a short testing period with a comparable speed of testing.

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[1] Engineer, Condenser and Heater Division, Worthington Pump & Machinery Corp., Harrison, N. J.

[2] Alfred H. White, Claude H. Leland, and Dale W. Button, "Determination of Dissolved Oxygen in Boiler Feed Water," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 697 (1936).

I feel that this length of time is not sufficient to indicate the true accuracy of the test method as a whole.

The dependence that can be placed on a test is in inverse ratio to the magnitude of its probable error as determined by statistical means over a period of time sufficiently long to include and take into account the element of physical and mental fatigue on the part of the operators.

I am very pleased to note that the equipment described in the Navy modifications of the Winkler Method includes the McLean flask and the flask nipple washer. In our use of this flask for the past nine years we have found it to be a most reliable method for the collection of samples. The flask nipple washer which we have used since 1940 is a practical necessity if test results of high accuracy are to be maintained.

There are some phases in the technique of the method described by Mr. Adams where errors in measurement, seriously affecting the accuracy of the test, are likely to occur.

One of these is the described practice of draining the contents of the reagent cup exactly to the upper surface of the stopcock on the McLean flask.

This is an extremely exacting and difficult procedure and presents a real possibility of error, both in measurement and in trapping air in the stopcock, which may then be introduced into the flask with the addition of the next reagent, thereby contaminating the sample.

In our use of the Navy Laboratory method we have modified the reagent cup by decreasing the bore and calibrating it between two etch marks in its straight length. By measuring the reagent additions in this manner the accuracy of measurement is increased to within 0.01 ml. of the desired and a seal of reagent is always present over the stopcock, effectively eliminating the possibility of trapping air in the flask.

The practice of controlling the flow of reagent to the sampling flask by throttling the upper stopcock places the sample under a partial vacuum and invites contamination by possible air leakage into the sample through the ground glass joint of the stopcock.

We use the reverse of this procedure in order to maintain a slight pressure in the sampling flask at all times prior to actual titration.

This latter practice is consistent with the authors' stated need for cooling the sample below room temperature in order to maintain positive pressure in the flask.

Our research has shown that occasionally the alkaline potassium iodide and manganous sulfate solutions remain in an unacidified state in the stopcock bore opposite the stopcock used for the addition of sulfuric acid. We have found that discarding the first 10 ml. of the sample drained from the end of the flask containing this trapped unfixed solution largely eliminates this possibility of sample contamination.

We feel that the omission of this simple procedure is conducive to occasional error of high magnitude and detracts from the inherent accuracy of the test.

The statistical analysis of results obtained from standardization test runs of 8 hr. duration has shown that the combined use of the flask nipple washer and the discarding of the unfixed reagents from the Winkler sample have doubled the accuracy of the test.

The application of the method to field testing is most satisfactory, requiring a minimum of one skilled and one semiskilled operator. The expense involved of transporting personnel and equipment is well worth while in view of the accuracy attainable.

MESSRS. R. C. ULMER,[3] J. M. DECKER,[3] AND J. M. REYNAR[3] (*presented in written form*).—The need for a method "for establishing the accuracy of an analytical determination of dissolved oxygen," as expressed by the authors, is real. Many of the misgivings regarding results of dissolved oxygen tests would be dispelled if a simple method were available for determining dissolved oxygen in absolute units.

The laboratory method presented, however, is not a very simple one and in our opinion has been complicated unnecessarily. Especially the method of obtaining samples of deaerated water of "constant oxygen content" and of adding the reagents is not particularly conducive to great accuracy. We have found that, for the determination of dissolved oxygen in absolute units, it is necessary to have oxygen-free water available. Also sampling under water eliminates the use of special equipment and facilitates the addition of reagents.

Moreover, in our opinion, based on both laboratory and plant experiences, the dead-stop end point gives equally as good results as the potentiometric titration described, and certainly is much simpler to use, as the exact end point is indicated and the plotting of a curve is not necessary.

Although the volume of the sample for the laboratory method is not mentioned, it apparently was approximately 500 ml. The value reported, for oxygen in reagents, 0.0104 ppm., therefore, does not agree with that of other investigators[4] who have reported values of approximately 0.020 ppm. when 2 ml. of each reagent was used for a 500-ml. sample. Presumably the reagents were not saturated with oxygen and they, therefore, would be likely to pick up oxygen during use, which would be a nuisance.

Potassium furoate has never been used in this laboratory so its merits as a stabilizer cannot be discussed, but borax has been found to be entirely satisfactory. The advantage of not having to prepare the stabilizer, as is the case with potassium furoate, is obvious. Solutions of sodium thiosulfate having a normality of 0.002 when stabilized with 3.8 g. of borax per liter, remain stable for several weeks.

MR. R. C. ADAMS[5] (*author's closure, by letter*).—Of the several points questioned by the discussors, some are matters of opinion or preference on which a reliable decision cannot be made without further trial. We recognize, for example, that the evaluation of the precision of any procedure will require a statistical number of successive determinations on a water of demonstrated, constant dissolved-oxygen content. We have not been able to arrange such a program and so have estimated the precision of our method from the apparent uniformity of results obtained from several hundred determinations made in many places on waters which were constant for periods of one to two hours. Mr. Sebald's opinion, in confirmation of our own, is most welcome.

There is no such thing as the "oxygen-free water" suggested by Mr. Ulmer so that any investigator of methods must be content with a "water of low and constant dissolved-oxygen content" as used by us. The "constant" dissolved-oxygen may be made less, but cannot all be removed by a more effective deaerator because the partial pressure of oxygen in the atmosphere above the water sampled cannot be reduced to zero in finite time.

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[3] Research Dept., The Detroit Edison Co., Detroit, Mich.

[4] J. D. Yoder and A. C. Dresher, *Combustion*, Vol. 5, p. 18 (1934).

[5] Assistant Head, Chemical Laboratory, U. S. Naval Engineering Experimental Station, Annapolis, Md.

Our value for oxygen dissolved in the Winkler reagents was obtained on aged, air-saturated reagents. White, Leland and Button[2] reported values which, for 500-ml. samples using 2 ml. of each reagent, correspond to 0.0102 ppm. However, none of their reagents were exactly the same as ours and 93 per cent of the oxygen was introduced by the manganous chloride solution while we employ a solution of manganous sulfate. Our value of 0.0104 ppm. therefore can scarcely be considered a corroboration of theirs, although it would appear to be essentially correct.

The dead-stop procedure and the use of borax as stabilizer may be as satisfactory as ours. It is questionable, however, if the approach of the dead-stop end point is readily apparent. The potassium furoate has been proved satisfactory by storage tests approaching three years duration. Most of Mr. Sebald's suggestions for minor changes in the procedure also have been tried, and some, the nipple washer for example, have been adopted. However, the procedure described is preferred and recommended after consideration of the merits and disadvantages of the several alternates.

APPLICABILITY OF THE SCHWARTZ-GURNEY METHOD FOR DETERMINING DISSOLVED OXYGEN IN BOILER FEEDWATER AND MODIFICATION OF THE METHOD TO MAKE IT ESPECIALLY APPLICABLE IN THE PRESENCE OF SUCH IMPURITIES AS ARE ENCOUNTERED IN POWER PLANTS

By R. C. Ulmer,[1] J. M. Reynar,[1] and J. M. Decker[1]

Synopsis

Since the Winkler method for determining dissolved oxygen in water was developed, various modifications have been suggested to increase its accuracy and sensitivity, especially in the presence of impurities. It has generally been assumed that the regular Winkler method gives results of sufficient accuracy for control purposes in the case of feedwater from plants using evaporated make-up. In case greater accuracy is desired, the Schwartz-Gurney modification involving certain refinements over the Winkler method has been used to some extent, but the results of this method as well as those of the Winkler are somewhat questionable when impurities that may react during the fixation of the sample are present. This paper presents a variation of the Schwartz-Gurney modification that has been found to give good results for dissolved oxygen under plant conditions, no difficulty having been encountered as a result of the reaction of impurities during fixation even when sulfite was present. Use of the method also makes possible the calculation of reducing and oxidizing agents present. Data are given that indicate that slight amounts of reducing and oxidizing agents, such as are present in power houses using evaporated make-up and sulfite boiler water treatment, do not interfere in the proposed test method. Confidence is claimed in the accuracy and general applicability of the Schwartz-Gurney modification, as employed.

Since the Winkler method was first proposed, numerous attempts have been made to increase the accuracy with which dissolved oxygen can be determined in water. No attempt will be made in this paper to discuss the pros and cons of this subject, but interested readers are referred to the bibliographies accompanying technical papers on the subject in recent years, such as the articles by Schwartz and Gurney (**1**),[2] White, Leland, and Button (**2**), and Daugherty (**3**).

On the basis of data given in these references and those obtained from the use of the proposed methods, it is rather generally agreed that in the case of pure water, such as condensed steam and feedwater in a power plant using evaporated make-up, the Winkler method gives results of sufficient accuracy to make possible the operation of the plant without serious corrosion. Nevertheless, there has been a desire on the part of many investigators for a method that will give more accurate results than the Winkler test. This desire has become much more pronounced in the past decade during which operating pressures and temperatures have increased markedly. In order to meet this need the Schwartz-Gurney double titration method (1) and the so-called evolution method by White, Leland, and Button (2) have been developed. These methods undoubtedly greatly increased the accuracy of the determination but, being complicated and difficult to carry out in power houses, they have never been widely accepted. This may be due in part to the fact that in most cases the publications dealing with the methods have contained extensive laboratory data and very little, if any, actual plant results or data. It is hoped that this paper, presenting some laboratory conclusions and the results of power house tests made with a method developed during some three years of plant experience and which is essentially the Schwartz-Gurney Method B, will lessen some of the apprehensions about this very practical and accurate method, the complexity of which has been generally overestimated.

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[1] Research Dept., The Detroit Edison Co., Detroit, Mich.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1266.

The need for a method more accurate than the Winkler became apparent in this company several years ago when it was noted that considerable amounts of corrosion products were accumulating in piping, condensers, and heaters in the four major power houses of the company, one of which was sulfite treated, even though the Winkler method indicated only traces or zero oxygen most of the time. The Schwartz-Gurney Method B using starch as an indicator was tried, but yielded unsatisfactory results because of the poor end point obtained with the very dilute titrating solutions needed by reason of the small amount of dissolved oxygen present. The "dead-stop" end point, applied to dissolved oxygen tests by Hewson and Rees (4), was found to give much better results and was adopted for use.

METHOD

The method as carried out in the plants by laboratory personnel is as follows:

Sampling:

Two 250-ml. and one 500-ml. samples are collected simultaneously by the use of the three-arm manifold suggested by Daugherty (3). The glass-stoppered bottles are placed in a bucket of such size that, when it is overflowing, the tops of the bottles are submerged several inches. Samples can be taken at 10 to 15 min. intervals by this method.

Reagents:

Manganous Sulfate solution and *Alkaline Iodide solution*, according to Am. Public Health Assn., Eighth Edition.

Concentrated HCl. C. P. reagent

Sodium thiosulfate, 0.002 *N*. Diluted from a stronger solution and containing 3.8 g. of borax per liter as stabilizer. The solution was adjusted so as to be exactly equivalent to the potassium iodate solution.

Potassium iodate solution, 0.002 *N*. Prepared from c.p. KIO_3 and containing 0.5 g. of $NaHCO_3$ per liter.

Procedure:

The three samples are collected as described. Using 1-ml. medicine droppers, 1 ml. each of manganous sulfate and alkaline iodide solutions are added in that order to the 500-ml. sample and to one of the 250-ml. samples to "fix" the oxygen. All three bottles are stoppered and the two to which reagents have been added are removed from the sampling bucket, inverted several times in order to mix the contents, and then

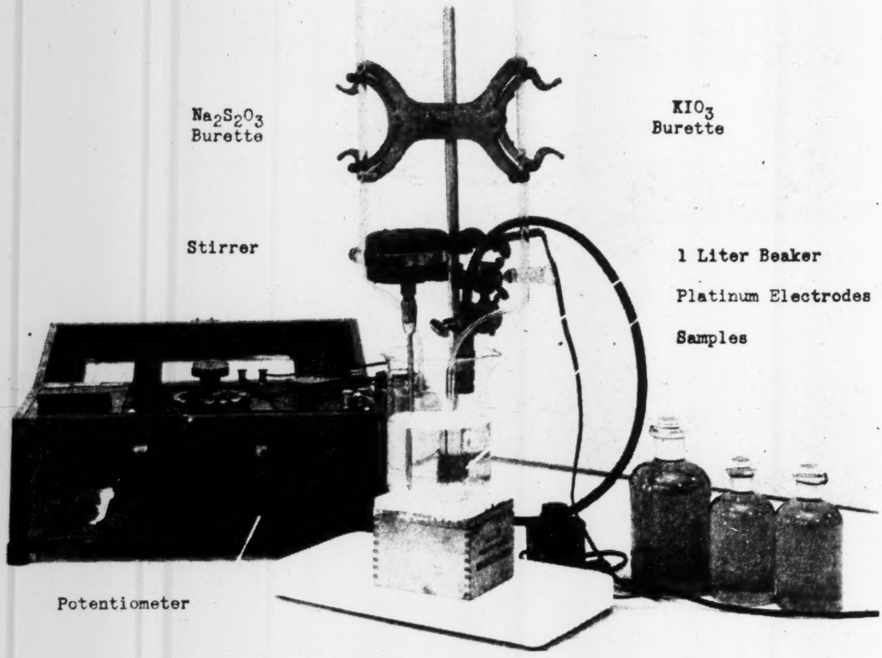
$Na_2S_2O_3$ Burette
KIO_3 Burette
Stirrer
1 Liter Beaker
Platinum Electrodes
Samples
Potentiometer

FIG. 1.—Titration Assembly for Dissolved Oxygen Determination.

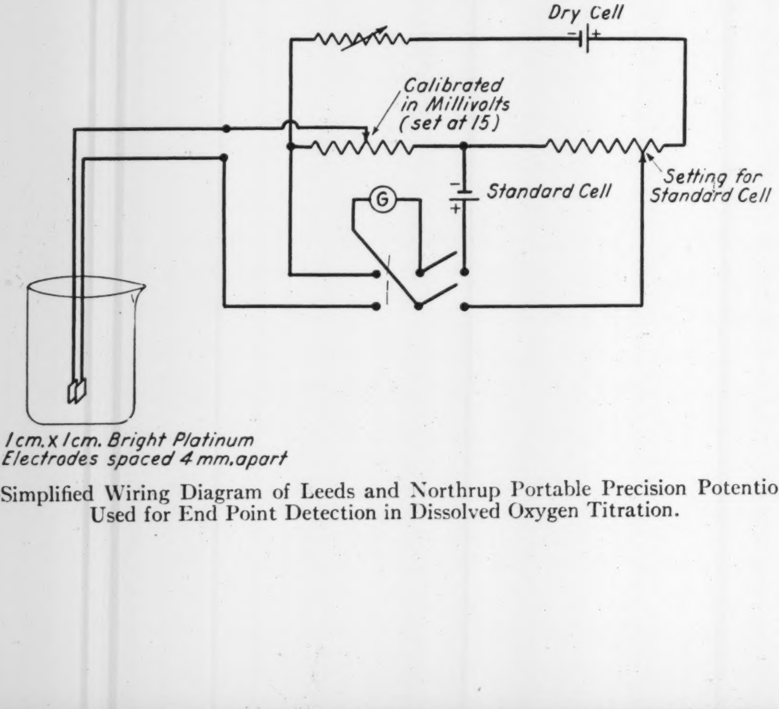
Dry Cell
Calibrated in Millivolts (set at 15)
Standard Cell
Setting for Standard Cell
G
1 cm. x 1 cm. Bright Platinum Electrodes spaced 4 mm. apart

FIG. 2.—Simplified Wiring Diagram of Leeds and Northrup Portable Precision Potentiometer as Used for End Point Detection in Dissolved Oxygen Titration.

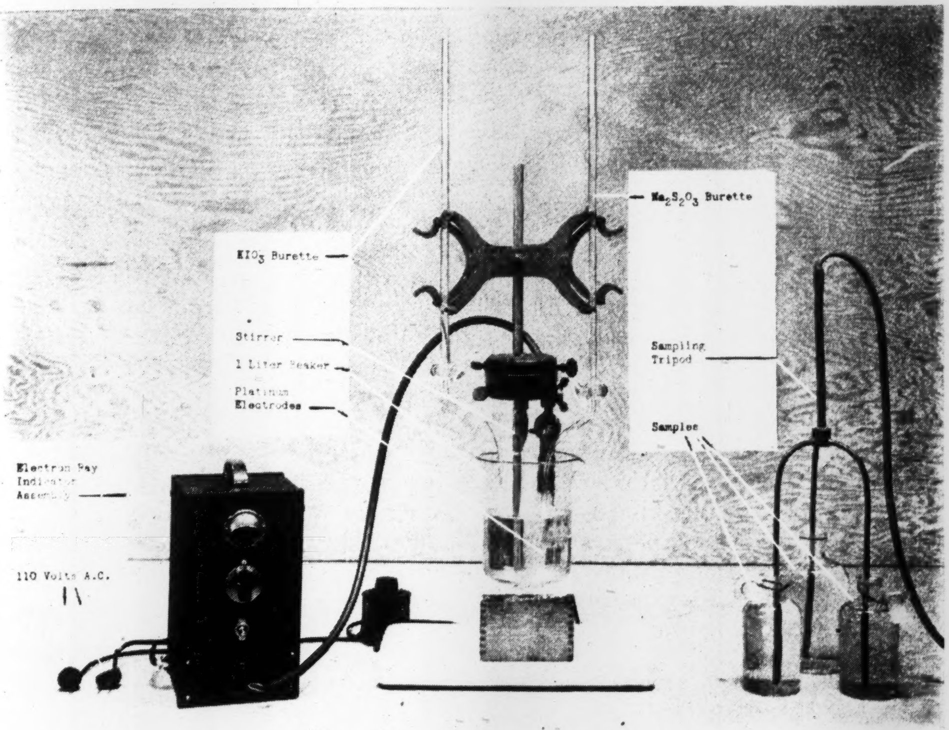
KIO_3 Burette
Stirrer
1 Liter Beaker
Platinum Electrodes
$Na_2S_2O_3$ Burette
Sampling Tripod
Samples
Electron Ray Indicator Assembly
110 Volts A.C.

FIG. 3.—Titration Assembly Using Electron-Ray Indicator Type Tube for Dissolved Oxygen Determination.

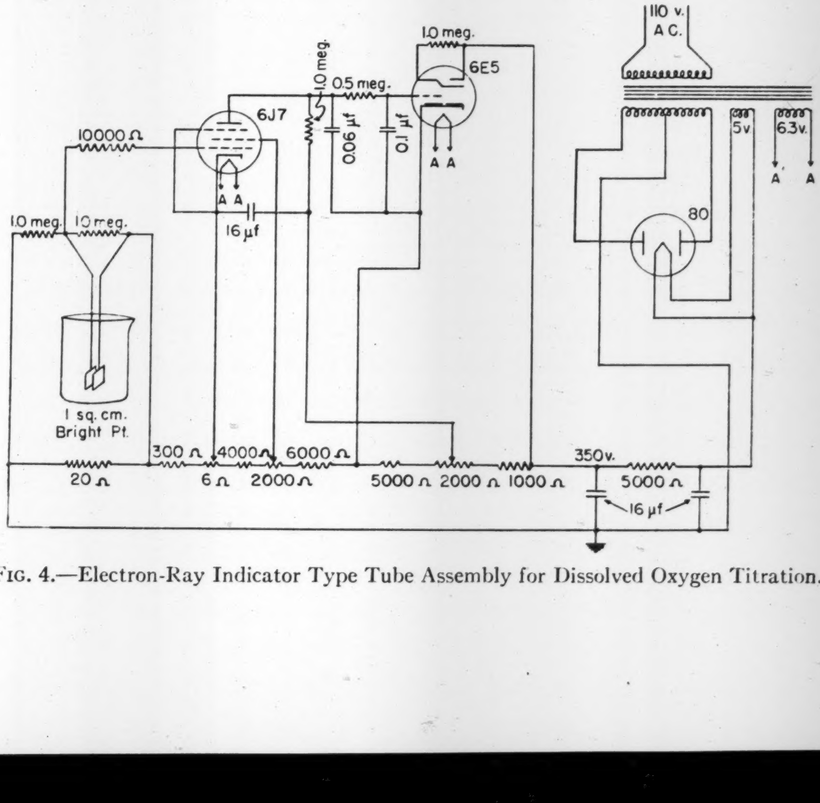
110 v. A.C.
1.0 meg.
6E5
1.0 meg.
0.5 meg.
6J7
10000 Ω
0.06 μf
0.1 μf
A A
A A
16 μf
1.0 meg.
1.0 meg.
5v
6.3v.
A A
80
1 sq. cm. Bright Pt.
300 Ω
4000 Ω
6000 Ω
350v.
20 Ω
6 Ω
2000 Ω
5000 Ω
2000 Ω
1000 Ω
5000 Ω
16 μf

FIG. 4.—Electron-Ray Indicator Type Tube Assembly for Dissolved Oxygen Titration.

returned to the bucket. After 5 min., 3 ml. of hydrochloric acid is added to each of the bottles to which the other reagents were added. The bottles are inverted several times for mixing and then removed from the bucket for titration.

The 500-ml. sample and the two 250-ml. samples, now combined, are titrated with the assembly shown in Figs. 1 and 2. The use of this apparatus in the titration of the samples is essentially the dead-stop end-point method described by Hewson and Rees (**4**). The sample to be titrated is placed in the beaker containing the electrode assembly (bare 1 by 1 cm. polished platinum) and a stirrer. If the solution is reducing, as indicated by the absence of deflection on the galvanometer, the titration is carried out with 0.002 *N* KIO_3 until the first permanent deflection of the galvanometer. If the solution is oxidizing, as indicated by deflection, 0.002 *N* thiosulfate is added until the galvanometer shows no deflection and then the solution is titrated with 0.002 *N* KIO_3. By always approaching the end point from the reducing side, a very sharp end point is obtained, easily discernible within one drop (0.05 ml.) of 0.002 *N* KIO_3 or 0.003 ppm. of oxygen. Under laboratory conditions, still greater sensitivity can be obtained, as stated by Hewson and Rees (**4**), and as confirmed by earlier work in this laboratory. Although 2 ml. of hydrochloric acid is usually called for in the Winkler test and its modifications, a poor end point was often obtained when this amount was used, and 3 ml. was found to give better results.

Although the apparatus referred to in Figs. 1 and 2 is to be recommended because it uses standard equipment available in most laboratories, it is somewhat tedious to use and is affected by vibration. Therefore an alternate assembly shown in Figs. 3 and 4 was devised. Instead of using a galvanometer, which is affected by vibration, this assembly uses an electron-ray indicator-type tube (magic eye) and an amplifier. The technique of titration is the same as that for the other assembly, the end point being indicated by the closing of the "eye" which is set at its maximum opening when the solution is reducing. The end point is easily discernible within one drop (0.05 ml.) of 0.001 *N* KIO_3 or 0.0015 ppm. of dissolved oxygen. Moreover, the apparatus is compact and ideal for plant use by unskilled operators.

The calculation of the dissolved oxygen and reducing or oxidizing agent present in the sample can best be understood if the various items involved in the titration of each of the two samples prepared for titration are considered.

Item	Sample A (500 ml. fixed)	Item	Sample B (250 ml. fixed + 250 ml. not fixed)
1	Dissolved oxygen in 500-ml. sample ($2x$)	1′	Dissolved oxygen in 250-ml. sample (x)
2	Contaminants in reagents 1 ml. manganous sulfate 1 ml. alkaline iodide 3 ml. HCl	2′	Contaminants in reagents 1 ml. manganous sulfate 1 ml. alkaline iodide 3 ml. HCl
3	Thiosulfate added if not already reducing (a')	3′	Thiosulfate added if not already reducing (a)
4	Reducing or oxidizing agent in 500-ml. sample (y)	4′	Reducing or oxidizing agent in 500-ml. sample (y)

The net thiosulfate for sample A minus that for sample B (item 1 − item 1′) is equivalent to the dissolved oxygen in 250 ml. of sample, since Items 2 and 2′, 3 and 3′, and 4 and 4′, cancel out. This can perhaps be seen more readily if it is approached mathematically.

For sample B (250 ml. fixed + 250 ml. not fixed):

Let x = thiosulfate equivalent of dissolved oxygen (that is in 250 ml.),

y = thiosulfate equivalent of the reducing or oxidizing agent inherently in a 500-ml. sample,

a = known thiosulfate added to give an excess, and
b = equivalent potassium iodate to back-titrate to the end point.

then for sample A (500 ml. fixed):

$2x$ = thiosulfate equivalent of dissolved oxygen (that is in 500 ml.),
y = thiosulfate equivalent of the reducing or oxidizing agent inherent in a 500-ml. sample,
a' = known thiosulfate to give an excess, and
b' = equivalent potassium iodate to back-titrate to the end-point.

Since at the end point, for example in the case of sample B, the quantity of oxidant equals the quantity of reductant

$$x + b = y + a \ldots\ldots\ldots (1)$$

For sample A

$$2x + b' = y + a' \ldots\ldots\ldots (2)$$

Subtracting Eq. 1 from 2

$$x = a' - a - b' + b \ldots\ldots (3)$$

which equals the milliliters of 0.002 N thiosulfate equivalent to the dissolved oxygen in 250 ml. of the sample. Therefore, $0.064 \cdot x$ equals ppm. of dissolved oxygen in the sample, where 0.064 is the factor for converting milliliters of 0.002 N KIO_3 to ppm. of dissolved oxygen.

Substituting for x in Eq. 1:

$$a' - a - b' + b + b = y + a$$
$$y = a' - 2a + 2b - b' \ldots (4)$$

or

$$y = a' - b' - 2\,(a - b) \ldots (5)$$

which equals the thiosulfate equivalent of the reducing or oxidizing agent inherent in 500 ml. of the sample. Therefore, $y \cdot \frac{0.064}{2}$ equals ppm. of reducing or oxidizing agent calculated as ppm. of dissolved oxygen. Since $a' - b'$ equals the net thiosulfate required for sample A (500 ml. fixed) and $a - b$ equals the net thiosulfate required for sample B (250 ml. fixed), the thiosulfate equivalent of the reducing agent can readily be obtained by substituting these values in Eq. 5. If y is positive, reducing agent is present; if negative, oxidizing agent is present. The data in Table I are typical of those taken during plant tests and demonstrate that the calculations are very simple.

DISCUSSION

The source of the water for the evaporators supplying the make-up for the power houses is the St. Clair River in the case of Marysville, and the Detroit River in the case of the three other plants. Although no specific tests have as yet been made to determine the exact nature and source of the reducing agent present when sulfite was not being used, there is good reason to believe that it results from sewage in the rivers. In this respect, it is of interest that the reducing agent is usually much higher immediately after a rain, when considerable surface water would be in the river, than during periods with little or no rainfall. The fact that the reducing agent varies considerably and that in a few cases oxidizing agents were present make the results of the Winkler test of somewhat questionable value for plant control purposes. Nevertheless, in the case of the plants listed, the Winkler test is used for control purposes, but the results obtained during an 8-hr. shift are checked twice a year by the proposed method. The plant results given in Table I represent such tests, and were obtained by the plant operators with the Winkler method, using starch indicator. Reasonable agreement was obtained in most cases unless a considerable amount of reducing or oxidizing agent was present.

In general, when sulfite was used, the amount of reducing agent was higher than when it was not used and the results of the plant tests were too low. Although no tests for sulfite in the condensate have been made, it is assumed that the increase in reducing agent during these periods was due largely to sodium sulfite carried over from the boilers, which occurrence has been reported by Hitchens and Purssell (5).

TABLE I.—PLANT DATA ON DISSOLVED OXYGEN TESTS OF BOILER FEEDWATER.

Plant[a]	Date	Time	Turbo-Generator	Na_2SO_3 Treatment Used	500-ml. Sample			250 ml. Fixed + 250 ml. Unfixed			0.002 N Thiosulfate for 250 ml., ml.	Dissolved oxygen, Schwartz-Gurney Method, ppm.[b]	Reducing Agent Expressed as O_2, ppm.	Plant Test, Winkler Method, O_2, ppm.
					0.002 N Thiosulfate, ml.	0.002 N Iodate, ml.	Net Thiosulfate, ml.	0.002 N Thiosulfate, ml.	0.002 N Iodate, ml.	Net Thiosulfate, ml.				
Marysville	7/ 3/42	1.25 p.m.	No. 4	No	1.00	0.80	0.20	1.00	1.05	−0.05	0.25	0.015	0.009	0.02
		1.50 p.m.	No. 4	No	1.00	0.85	0.15	1.00	0.95	0.05	0.10	0.006	0.001	0.03
		3.10 p.m.	No. 4	No	1.00	0.60	0.40	1.00	0.80	0.20	0.20	0.013	0	0.01
	10/14/42	1.25 p.m.	No. 6	No	0	0.35	−0.35	0	0.40	−0.40	0.05	0.003	0.014	0
		2.40 p.m.	No. 6	No	0.50	0.50	0	0.50	0.60	−0.10	0.10	0.006	0.006	0
Conners Creek	2/13/42	11.45 a.m.	No. 9	Yes	0.50	0.85	−0.35	0.50	1.20	−0.70	0.35	0.021	0.032	0.01
		12.50 p.m.	No. 9	Yes	0.50	1.05	−0.55	0.50	1.25	−0.75	0.20	0.013	0.028	trace
		3.45 p.m.	No. 9	Yes	0.50	1.00	−0.50	0.50	1.30	−0.80	0.30	0.019	0.033	trace
	1/26/43	10.35 a.m.	No. 13	No	1.00	0.25	0.75	0.50	0.15	0.35	0.40	0.025	0.001	....
		1.15 p.m.	No. 13	No	0.90	0.30	0.60	1.00	0.70	0.30	0.30	0.019	0	....
		2.50 p.m.	No. 13	No	1.00	0.28	0.72	0.50	0.14	0.36	0.36	0.023	0	....
	2/17/43	1.55 p.m.	No. 13	No	0	0.15	−0.15	0	0.15	−0.15	0	0	0.004	0.15
		2.45 p.m.	No. 13	No	0	0.20	−0.20	0	0.34	−0.34	0.14	0.009	0.014	trace
	4/ 3/43	1.20 p.m.	No. 13	Yes	0.75	0.03	0.72	0.50	0.32	0.18	0.54	0.032	0.011	....
		2.20 p.m.	No. 13	Yes	0	0.05	−0.05	0	0.10	−0.10	0.05	0.003	0.004	....
Delray	3/13/42	.....	No. 13	No	0.50	0.43	0.07	0.50	0.45	0.05	0.02	0.001	−0.001	0
		.....	No. 14	No	0.50	0.45	0.05	0.50	0.50	0	0.05	0.003	0.001	0
	2/11/43	3.15 p.m.	No. 13	No	0.25	0.15	0.10	0.25	0.30	−0.05	0.15	0.010	0.012	trace
		3.30 p.m.	No. 13	No	0.25	0.10	0.15	0.25	0.32	−0.07	0.22	0.014	0.009	0.01
Trenton Channel	2/24/42	10.20 a.m.	No. 5	No	1.00	0.80	0.20	0.50	0.30	0.20	0	0	−0.006	0.02
		11.15 a.m.	No. 4	No	0.50	0.30	0.20	0.50	0.40	0.10	0.10	0.006	−0.009	0.01
	7/24/42	2.05 p.m.	No. 5	No	0.50	0.50	0	0.50	0.50	0	0	0	0	0.02
		3.10 p.m.	No. 5	No	0.50	0.46	0.04	0.50	0.48	0.02	0.02	0.001	0	0
	2/25/43	12.45 p.m.	No. 4	No	1.00	0.70	0.30	0.50	0.52	−0.02	0.32	0.020	0.012	0.01
		3.30 p.m.	No. 4	No	0.50	0.05	0.45	0.25	0.20	0.05	0.40	0.025	0.015	0.02

[a] The following are the steaming conditions at the plants in question:

Plant	Throttle Pressure, psi.	Throttle Temperature, deg. Fahr.
Marysville	275	650
Conners Creek	600	825
Delray	815	900
Trenton Channel	375	700

[b] Owing to slight variations in the volume of the bottles used, the factor varies somewhat from the value of 0.064 for a 250-ml. sample. In all cases the total sample was titrated and a volume correction applied, rather than to titrate a measured volume of 250 ml.

Regardless of the nature of the reducing agent, apparently it did not interfere in the modified Schwartz-Gurney test procedure used in this work. This may be due to the fact that only a small amount was present, inasmuch as numerous investigators (**6, 7, 8**) have found

TABLE II.—INTERFERENCE OF REDUCING AGENT WITH TEST RESULTS OBTAINED AT CONNERS CREEK PLANT.

Date	Time	Turbo-Generator	Na_2SO_3 Treatment Used	Time After Sampling when "Fixed"	Time After Fixing when Titrated	Order[b] of Adding Reagents	Reducing Agent as O_2, ppm.	500-ml. Sample				250 ml. fixed + 250 ml. unfixed,			Thiosulfate for 250 ml., ml.	Dissolved Oxygen, Schwartz-Gurney Method, ppm.	Remarks
								0.002 *N* Thiosulfate, ml.	0.002 *N* Iodate, ml.	Net Thiosulfate, ml.	Dissolved Oxygen, Winkler Method, ppm.	0.002 *N* Thiosulfate, ml.	0.002 *N* Iodate, ml.	Net Thiosulfate, ml.			
March 12, 1943	10.55 a.m.	No. 13	No	Immediately[a]	Immediately	Regular	0.003	1.50	0.50	1.00	0.030						
		No. 13	No	5 min.	Immediately	Regular	0.003	1.50	0.53	0.97	0.029						
		No. 13	No	25 min.	Immediately	Regular	0.003	1.50	0.51	0.99	0.030						
		No. 13	No	1 hr.	Immediately	Regular	0.003	1.50	0.52	0.98	0.029						
		No. 13	No	Immediately[a]	Immediately	Regular	0.003	1.50	0.50	1.00	0.030						
		No. 13	No	Immediately[a]	10 min.	Regular	0.003	1.50	0.45	1.05	0.031						
		No. 13	No	Immediately[a]	25 min.	Regular	0.003	1.50	0.45	1.05	0.031						
		No. 13	No	Immediately[a]	1 hr.	Regular	0.003	1.50	0.45	1.05	0.031						
March 24, 1943	11.45 a.m.	No. 13	Yes	Immediately	Immediately	Regular	0.019	0.50	0.16	0.34		0.00	0.15	−0.15	0.49	0.029	
		No. 13	Yes	5 min.	Immediately	Regular	0.017	0.50	0.20	0.30		0.00	0.13	−0.13	0.43	0.026	
		No. 13	Yes	23 min.	Immediately	Regular	0.020	0.50	0.07	0.43		0.00	0.12	−0.12	0.55	0.033	
		No. 13	Yes	1 hr.	Immediately	Regular	0.018	0.50	0.15	0.35		0.00	0.13	−0.13	0.48	0.029	
April 23, 1943	10.55 a.m.	No. 8	Yes	Immediately	Immediately	Regular 1-2-3	0.021	0.00	0.20	−0.20		0.00	0.45	−0.45	0.25	0.015	
	11.35 a.m.	No. 8	Yes	....	....	2-3-1	....	0.00	0.95	−0.95		0.00	0.98	−0.98			½ hr. interval between (2) and (3)
	1.00 p.m.	No. 8	Yes	....	....	2-3-1	....	0.00	0.92	−0.92		0.00	0.98	−0.98			5 min. interval between (2) and (3)
	1.20 p.m.	No. 8	Yes	....	....	3-2-1	....	0.00	0.98	−0.98		0.00	0.98	−0.98			Reagents added in quick succession
	1.45 p.m.	No. 8	Yes	....	....	3-2-1	....	0.00	1.05	−1.05		0.00	1.08	−1.08			Reagents added in quick succession
	2.15 p.m.	No. 8	Yes	Immediately	Immediately	1-2-3	....	0.50	0.82	−0.32		0.00	0.70	−0.70	0.38	0.023	
	2.40 p.m.[c]	No. 8	Yes	....	....	3-2	....	0.00	0.87	−0.87							Reagents added in quick succession
	2.40 p.m.[c]	No. 8	Yes	....	....	2-3	....	0.00	0.88	−0.88							Reagents added in quick succession
	2.40 p.m.[c]	No. 8	Yes	....	....	3 & KI	....	0.00	0.85	−0.85							Reagents added in quick succession
	3.15 p.m.[c]	No. 8	Yes	....	....	3 & KI	....	0.00	0.55	−0.55							Reagents added in quick succession
	3.15 p.m.[c]	No. 8	Yes	Immediately	Immediately	1-2-3	0.013	0.00	0.47	−0.47		0.00	0.45	−0.45	0.00[d]	0.000	

[a] Refers to time that manganous sulfate and alkaline iodide were added. In all cases, the HCl was added 5 min. after the other reagents.

[b] Reagents are referred to as follows: (1) 1 ml. manganous sulfate, (2) 1 ml. alkaline iodide, and (3) 3 ml. of concentrated hydrochloric acid.

[c] Steam sample.

[d] Actually this value would be −0.02, which amount is within the limit of error of the titration.

it necessary to correct for reducing agent. In Table II are given data obtained in a series of tests to determine whether the proposed method could be expected to give accurate results in the presence of reducing agent. The data shown for March 12, 1943, were obtained in order to determine whether it was necessary to fix and titrate the samples soon after being taken. As indicated, allowing duplicate samples to stand for periods up to 1 hr. before and after fixing, had little or no effect on the amount of oxygen obtained. The modified Schwartz-Gurney data on March 24, 1943, also indicate that the samples may be allowed to stand in flowing sample water for periods up to 1 hr. before fixing, without any effect on the results. From these data it may be concluded that the dissolved oxygen and reducing agent do not react in neutral solutions at room temperature with sufficient speed to interfere with the accuracy of the test.

The data shown for April 23, 1943, were obtained largely to determine whether the dissolved oxygen and reducing agent might be expected to react in an alkaline solution such as would be present when the samples were being fixed. These data (11.35 a.m., 1.00 p.m., and 2.40 p.m. samples) indicate that no significant reaction occurs in periods up to ½ hr., as is indicated by the fact that there is no decrease in the reducing action of the sample on standing with only alkaline iodide present. It is probable, therefore, that the values for dissolved oxygen represent all that was present in the samples. Furthermore, the data for 2.40 p.m., April 23, 1943, indicate that the reducing agent is actually in the sample and not in the reagents: for the reducing agent could be titrated directly without adding any of the reagents.

Some data were obtained in the work that indicate that the oxygen and reducing agent react rather rapidly when heated to temperatures of 300 to 500 F., even in neutral solution. For example, the dissolved oxygen content of feedwater was decreased from 0.030 to 0.010 ppm. in passing through the feedwater heaters. The reducing agent decreased accordingly. Further work is planned on this subject to determine whether there is any relation between this phenomenon and the formation of deposits in water heaters and piping.

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DISCUSSION

MR. J. F. SEBALD[1] (*by letter*).—It is gratifying to the designers and manufacturers of deaerating equipment to know that the engineering staff and operators of large power plants are recognizing the benefits to be derived from the use of refined dissolved oxygen test methods for investigation of performance of equipment and for the interpretation of control tests. For a long time the manufacturers of deaerating apparatus have been desirous of having the performance of their precisely designed equipment measured by accurate and reliable test methods, and it is a source of satisfaction to know that the authors have found oxygen test methods of sufficient importance to devote so much time to their development and application.

The author's use of the electron-ray indicator type tube for determining the end point of the oxidation reduction titration appears to have a definite advantage over the usual method employed. It not only eliminates the use of the rather inconvenient calomel half cell, but also eliminates the necessity of recording considerable test data. Although we feel that the use of the "magic eye" may be advantageous we cannot agree that the sampling method employed is entirely satisfactory. Our experience has showed that the McLean flask is very much more reliable, effectively eliminating air contamination of the samples during the addition of the Winkler reagents.

Whether any of the refined test methods, employing modifications for interference correction, will effectively compensate for sodium sulfite is, in our opinion, questionable. Our work in this field substantiates the findings of Haase, Wesly, and Thierault and McNamee, references 6, 7, 8 of the bibliography, with even smaller amounts of sodium sulfite present than that reported by the authors. We have, however, confirmed by field tests that the reducing agents (principally sodium sulfite) in surface condenser condensate vary directly with the sulfite concentration in the boilers. We have also observed on the same group of tests that the net dissolved oxygen in the condensate, when measured by an interference-corrected oxygen test method varies inversely with the sulfite concentration in the boilers, indicating that the correction for sulfite was incomplete.

Inasmuch as there is such a diversity of opinion among advocates of each of the three generally accepted precision test methods, namely, Schwartz-Gurney method B, biniodate modification of the Winkler method,[2] and the Navy Laboratory modification of the Winkler method (free iodine method)[2] regarding their effectiveness in correcting for interference, it would seem that an evaluation of their effectiveness should be made in the interest of the field of deaeration.

Much credit is due Messrs. Ulmer, Reynar, and Decker for their comprehensive discussion and for their contribution to the science of precise dissolved oxygen testing.

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[1] Engineer, Condenser and Heater Division, Worthington Pump & Machinery Corp., Harrison, N. J.

[2] Robert C. Adams, Robert E. Barnett, and Daniel E. Keller, Jr., "Field and Laboratory Determination of Dissolved Oxygen." See page 1240.

MESSRS. R. C. ULMER,[3] J. M. REYNAR,[3] AND J. M. DECKER,[3] (*authors' closure, by letter*).—Although only one official discussion has been submitted for this paper, it has been very gratifying to the authors to find that the paper has created much interest in the method. In fact, several individuals are building the end point apparatus and several of the instrument companies are interested in the apparatus. It is hoped that the apparatus can be made available commercially as many power companies are not equipped to build such apparatus. With respect to assembling the apparatus, some difficulty was encountered by persons building it in adjusting the circuit so as to obtain the desired sensitivity. For this reason the circuit shown in the paper as preprinted was modified slightly as shown in the present Fig. 4. It is thought that with this modification no difficulty will be encountered in assembling and adjusting the circuit so as to obtain the desired sensitivity.

Mr. Sebald's comments regarding the method in general, are gratifying. With respect to the use of the McLean tube we could find no way to make its use applicable to routine use in the power houses. We feel that the method described by Daugherty as used in our work is more convenient to use and we feel that there is no contamination of the sample on adding reagents.

With respect to correction for sodium sulfite present in the sample, we feel that the method as carried out compensates for all that is present at the time the test is carried out, but whether all of the oxygen present in the deaerator or condenser still remains at that time is a question. In other words, much more oxygen may have been present but reacted with sulfite prior to sampling and testing. With respect to corrosion this is of little if any import but with respect to efficiency of deaeration it would be desirable to know the concentration inside the deaerator or condenser.

With respect to Mr. Sebald's suggestion that the various methods for determining dissolved oxygen be evaluated in the interest of the field of deaeration, this can be done in a general way, but what may be applicable in the case of stationary power plant tests may not be applicable in the case of ships and the reverse may be true in many cases.

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SYMPOSIUM ON THE IDENTIFICATION OF WATER-FORMED DEPOSITS, SCALES, AND CORROSION PRODUCTS BY PHYSICO-CHEMICAL METHODS

INTRODUCTION

By H. C. Miller[1]

When in 1942 Committee D-19 on Water for Industrial Uses expanded the scope of some of its subcommittees to include methods for sampling and analysis of water-formed deposits, scales, and corrosion products, it was bringing together practically indissociable aspects of the industrial use of waters.

In transferring a natural water from its normal habitat to an industrial use, a struggle between factors of heredity and environment develops. To use water satisfactorily in an industrial process, examination of such hereditary traits as composition and amount of dissolved solids and gases, and environmental factors, such as temperature and pressure, is necessary. These must be intelligently evaluated, and when found to be incompatible, must be brought into proper balance. This is usually most easily accomplished by altering hereditary traits, using proper methods of water conditioning. When proper balance has not been attained, there usually develop clues in the form of what R. E. Hall has very aptly called the "fingerprints which water leaves behind," and which we immediately recognize as scales and deposits. In order that all the onus does not fall upon individuals responsible for corrective water treatment, it must be pointed out that a great many of worst cases of scale and deposit formation are the result of changes or variations in environmental factors of operation which are beyond their control.

The absence of "fingerprints," and this term includes those evidences of corrosion not accompanied by deposits, is usually a good indication that all factors in the system are in balance. When "fingerprints" are found, the fund of information they contain and imply is all too frequently but partially developed and utilized. That they can be made to yield data which indicates the mechanism of their formation and the means of preventing their recurrence, by utilizing supplemental methods of examination, is discussed in the papers comprising this symposium.

In the past the determination of composition, method of formation, and means of prevention of scales has been based largely on data obtained from macrochemical analysis. That this information was insufficient, unsatisfactory, and frequently misleading has long been recognized. Scales and deposits containing appreciable amounts of calcium, magnesium, phosphates, silica, iron, and copper are formidable combinations to analyze with accuracy. Where the state of oxidation of iron and copper is of importance, chemical analysis is even more uncertain. Even in those cases where reasonably accurate analyses have been obtained, interpretation of results is still a problem which is not appreciably simplified by the use of hypothetical combinations.

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Probably no data are as valuable in the study of scales and their formation as a knowledge of the exact form in which the constituents exist. Information of this type, within the limitations to be discussed in the following papers, can be obtained from petrographic and X-ray analysis. The ability of these methods to distinguish the form in which a polymorphous substance exists is of great value, and by utilizing the data available on transition points of these substances it is frequently possible to state with considerable accuracy the conditions of temperature and pressure which must have existed for their formation.

The water-conditioning engineer has not been slow to recognize the value of these methods, and in recent years has used them to considerable extent. Examination of turbine blade deposits by X-ray diffraction methods is not at all unusual. Silica, long a troublesome and puzzling constituent of such deposits has been found in the form of quartz, and, as such, apparently crystallized from the vapor phase. Information of this type may aid inestimably in current research on this problem. A subsidiary of a large chemical company engaged in acid cleaning of boilers and heat exchange equipment uses the X-ray to determine the nature of material to be removed in order that proper selection of acid and inhibiter may be made.

To date there are few references in the literature on the use of physico-chemical methods for examination of water-formed scales, deposits, and corrosion products. Much can, and we hope will, be written on the subject.

The papers which follow stress the use of the petrographic microscope and the X-ray although other, and in many cases, equally valuable methods such as the spectrograph may be used.

The type of information which may be obtained from X-ray diffraction studies of scales and corrosion deposits, and a discussion of the advantages and disadvantages of this method of examination is presented in the paper by Imhoff and Burkardt. A description of the problems encountered when investigating a scale sample and a discussion of various methods used to identify its components is given by Holmes and Walker. Partridge, Scott, and Morrison offer a diagnosis of typical water problems at a mythical power plant which illustrates the necessity for supplemental methods of examination, particularly on complex and unusual deposits.

TABLE I.—OPERATING HISTORY SUPPLIED TO LABORATORIES COOPERATING IN ROUND-ROBIN TESTS.

SAMPLE A

Cross-section of heating coil from commercial water heater using lime-softened river water.

Typical analysis of softened water as supplied by municipal water works:

	ppm.
Dissolved solids	150
Alkalinity	33
Calcium	15
Magnesium	6
Sulfate	37
Chloride	16
Silica	5
Iron oxide	0.02
Alumina	0.01

Temperature of water in hot-water system not known.

Coil became plugged after about four years of operation.

SAMPLE B

Hard deposit from straight inclined tubes of 450-psi. cross-drum boiler operated at 500 per cent of nominal rating.

Boiler-water conditions are indicated by the following analytical values for a spot sample and upper and lower limits from plant control records during period in which deposit developed:

	Spot Sample, ppm.	Range of Concentrations, ppm.
Hydroxide (OH)	383	200 to 400
Phosphate (PO_4)	70	30 to 125
Sulfate (SO_4)	1350	680 to 1350
Chloride (Cl)	128	70 to 130
Silica (SiO_2)	152	...
Aluminum (Al)	0	...

Boiler had operated for years at an output of 50,000 lb. of steam per hour without developing deposits. About five months after output was increased to 70,000 lb. per hr., deposits of hard scale were found generally throughout tubes.

Specimen was taken from upper-row tube near downtake header; this is a relatively cool region located in the fourth gas pass.

After deciding upon the subject of this symposium, and developing its context.

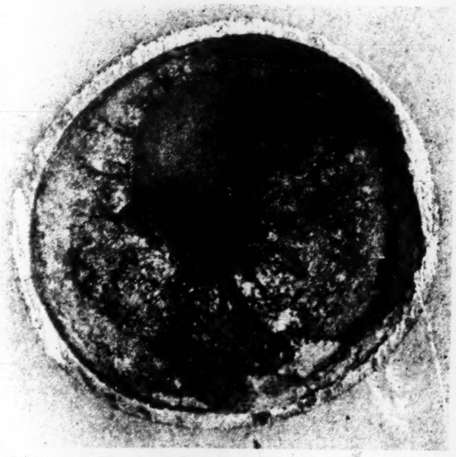

FIG. 1.—Typical Cross-Section of Sample A.

forwarded with the samples. This information appears in Table I, and photographs of typical sample portions are shown in Figs. 1 and 2.

The results received from the cooperating laboratories indicate three general types of tests were used in examining the two samples: (1) macrochemical analysis, (2) petrographic examination, and (3) X-ray diffraction studies. It was originally intended to demonstrate also the use of microchemical and spectrographic methods; however, neither of the samples submitted required the use of these methods nor does it appear that additional pertinent information would have been obtained therefrom.

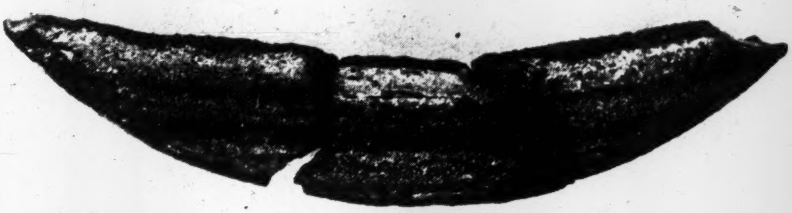

FIG. 2.—Typical Section of Sample B.

In discussing the results of the round-robin test, the following comments are those of the author only and do not constitute the opinion of Committee D-19 nor do they infer concurrence of of that group. While some of the comments are critical, it is hoped that they are objectively so.

Study of the results of the macrochemical analysis, which appear as Table II, focuses attention on two points. There is only partial agreement on the items selected for analytical determination. As this is largely a matter of personal preference and usually reflects the analysts opinion of which items are most likely to supply pertinent information we have no particular comment to make. The second point, which is

your committee believed a little demonstration of the application of these newer techniques might be of interest. They therefore enlisted the cooperation of five laboratories in a round-robin test program. Two samples of deposits, typical of those most of us frequently encounter, were obtained. Portions of these samples were sent to each of the laboratories with the instruction that they be submitted to macrochemical analysis and to any other special method of examination at their disposal. Upon the completion of the tests it was requested that data obtained and opinion as to the composition and probable mechanism of scale formation be submitted to the committee. To assist in reaching these decisions, as much data as was available on the nature of the raw water supply and on plant operating conditions was readily apparent, is the discrepancy in values obtained on identical items. In partial explanation of this it should be pointed out that no attempt was made in preparing the samples to ensure uniformity of composition by pulverizing and quartering them. It was decided to leave the samples in as nearly the state of their occurrence as was possible because it was known that X-ray and petrographic examinations were to be made. However, as there is good agreement between all laboratories on one or two items of analysis, we are of the opinion that the sample portions were pretty much alike, and that the discrepancies referred to are as much the result of the use of different analytical procedures as to variations between samples. (The analyses submitted by laboratory X are not necessarily considered "out of line" as they were made on mechanically separated portions of the sample, and are not composite.) This is somewhat borne out from another angle—that of the totals obtained. As these were re-

TABLE II.—MACROCHEMICAL ANALYSIS.

	Sample A						Sample B					
	Laboratory V	Laboratory W	Laboratory X		Laboratory Y	Laboratory Z[d]	Laboratory V	Laboratory W	Laboratory X		Laboratory Y	Laboratory Z
			a	b					a	b		
Loss on ignition	7.2		4.3[c]	8.1[c]	10.1	9.10[e]	6.2	...	4.5	6.6	4.6	3.40[e]
SiO_2	34.8	34.00	30.3	42.0	35.5	34.65	39.8	35.00	35.1	42.7	36.8	35.29
R_2O_3	...	...	2.3	3.4	3.8	...		...	...		...	...
Fe_2O_3	1.5	7.20	...	...	1.9	3.84	24.5	50.40[f]	34.5	20.0	22.6	37.88
Fe_3O_4	...		...	...	...	...		...	...	...	...	14.06
Al_2O_3	...	0.85	...	...	1.9	...	13.9	11.60	5.5	9.2	9.3	...
CaO	5.3	4.12	6.2	0.8	3.5	5.50	4.5	4.00	1.2	2.4	2.7	2.50
MgO	44.4	42.10	41.5	43.1	43.2	43.30	1.4	2.85	1.8	1.3	1.9	0.86
CuO	4.0	2.39	...	...	...	1.60	...	0.48	...	...	...	...
Cu_2O	...	...	9.0	0.8	2.8	...	...	...	...	...	...	...
CO_2	2.3	...	5.0	2.3	1.8	1.20	...	...	...	...	0.2	...
SO_3	...	0.16	0	0	0.1	...	...	0.47	0.6	0.7	0.3	tr.
P_2O_5	...	0.40	0	0	0	...	2.8	3.70	2.2	2.9	2.4	1.34
Na_2O	...	...	...	...	...	0.20	6.8	...	14.2	13.4	8.6	4.40
MnO	...	...	...	...	...	0.03	...	...	...	...	...	0.04

[a] Layer next to tube.
[b] Subsequently deposited layer.
[c] Calculated from loss at 400 C. plus calculated increase due to oxidation of Cu_2O to CuO.
[d] Sample dried at 105 C. before analysis.
[e] Reported as organic matter.
[f] Total iron.

TABLE III.—INTERPRETATION OF MACROCHEMICAL ANALYSIS.

	Sample A	Sample B
Laboratory V	No comment on basis of macrochemical analysis.	No comment.
Laboratory W	Bicarbonates are precipitating in the lines as a result of heating, with subsequent blockage. High silica content of scale surprising on basis of raw water analysis furnished. Believes a sodium silicate feed may have been used.	The deposition is probably complex silicates. From high iron content it might be concluded that corrosion is being experienced but definite conclusions not warranted without more complete knowledge of entire plant set-up. Iron might also have been removed by sampling.
Laboratory X	It is possible to infer that major constituent of each layer is an unknown magnesium silicate and that calcium carbonate is present in significant amount in layer next to tube. Attention called to difference in ratio of MgO to SiO_2 in the two layers.	In absence of supplemental information from microscopic or X-ray identification of actual constituents, it would be difficult to interpret these analyses conclusively.
Laboratory Y	No comment.	No comment.
Laboratory Z	Analysis is peculiar and one would hardly expect to find this type of scale under conditions mentioned.	Analysis is peculiar and one could hardly expect to find this type of scale under condition mentioned.

ported in only one case, they have been omitted from all.

It need hardly be pointed out to anyone familiar with scales and deposits that the combination of substances present in these samples is such as may make accurate analysis difficult. In commenting on the discrepancies, we are casting no reflections on the cooperating laboratories, but rather we emphasize the point to demonstrate the need for a dependable standard method of analysis for simple, as well as complex, scales and deposits. To the best of our knowledge, no such method exists. Frankly, some of the members of the committee harbored the morbid hope that just such discrepancies would arise from this little experiment to draw attention more emphatically to the need for the methods now in preparation.

TABLE IV.—PETROGRAPHIC ANALYSIS

	Sample A	Sample B
Laboratory V	$CaCO_3$ minor constituent. Major constituent not identified, crystals too small for microscopic examination.	Opaque granules probably high in Fe_2O_3. Analcite present. An iron silicate is indicated because of high index of refraction of one of the constituents.
Laboratory X	The major constituent of this deposit is an unidentified magnesium silicate. In the layer closer to the tube wall, calcium carbonate is also present in considerable amount, but subsequently deposited layer contains only a very small amount of this constituent. By chemical tests under the microscope, a copper compound was indicated in the layer next to the tube wall, but not in the other layer. The calcium carbonate is in the form of calcite; the magnesium silicate appears amorphous under the microscope.	Analcite definitely and sodium iron silicate probably major constituents. In the lighter colored outer layer there is more analcite than in the underlying darker colored material which is predominantly a crystalline material of high refractive index believed to be sodium iron silicate. Iron oxide is present throughout both layers. The sodium iron silicate is not definitely idenfied but is believed to be acmite ($Na_2O \cdot Fe_2O_3 \cdot 4\,SiO_2$).

Now as to interpretation of the composition and method of formation of those scales on the basis of macrochemical analysis, the best guesses of the cooperating laboratories are recorded in Table III. That these are frankly guesses is not only admitted by the cooperating laboratories but is easily understandable to those of us making similar tests. In the case of sample A, there does not seem much reason to doubt the presence of magnesium silicate, but there is temptation, using hypothetical combinations, to include other magnesium compounds such as the hydroxide. That this would be erroneous will be shown later. Analyses of sample B, however, have us entirely up in the air. Here is a combination of ions to challenge the most ingenious calculator of the hypothetical. If these analyses were made as part of actual plant investigations, it is unthinkable that any of the laboratories would have stopped working at this point; however, we think it evident that little information of value in interpreting actual composition or mechanism of formation of these scales has been obtained from the macroanalysis.

Let us go a little further in our investigation and submit these samples to petrographic examination. The two laboratories who did this obtained the information in Table IV. Both laboratories identify the presence of calcium carbonate, in the form of calcite, as a minor constituent of sample A, but the major constituent is not identified because it appears amorphous under the microscope. Sample B, however, responds more generously to petrographic technique. Both laboratories definitely detect analcite and an iron silicate as major constituents and further state that the sample is interspersed throughout with iron oxide particles. This information, while less detailed, is more specific than that obtained from macroanalysis, as it gives us data on the exact form in which some of the ions in the sample are combined. That this is a great advantage will be seen later when we come to interpretation of results. Further comment on the merits of either petrographic or X-ray methods of examination need not be made here as this is so well covered in the papers.

TABLE V.—X-RAY DIFFRACTION ANALYSES.

	Sample A	Sample B
Laboratory V	(Mg_2SiO_4) .... 80 per cent $CaCO_3$ .... 20 per cent	Fe_2O_3 (Hematite) .... 25 per cent Fe_3O_4 (Magnetite) .... 25 per cent $Na_2O\cdot Al_2O_3\cdot 4SiO_2\cdot 2H_2O$ (Analcite) 30 per cent $Na_2O\cdot Fe_2O_3\cdot 4SiO_2$ (Acmite) .... 20 per cent
Laboratory X	Layer next to tube Mg_2SiO_4 (Forsterite) major constituent $CaCO_3$ (Calcite) secondary constituent Cu_2O (Cuprite) small amount also present Subsequently deposited layer shows only magnesium silicate but this is present both as Forsterite (Mg_2SiO_4) and as Serpentine ($3MgO\cdot 2SiO_2\cdot 2H_2O$). The Serpentine is probably present in the greater amount.	Outer layer $Na_2O\cdot Al_2O_3\cdot 4SiO_2\cdot 2H_2O$ (Analcite) major constituent $Na_2O\cdot Fe_2O_3\cdot 4SiO_2$ (Acmite) considerable Fe_2O_3 (Hematite) minor constituent Fe_3O_4 (Magnetite) minor constituent Layer near tube surface $Na_2O\cdot Al_2O_3\cdot 4SiO_2\cdot 2H_2O$ (Analcite) major constituent $Na_2O\cdot Fe_2O_3\cdot 4SiO_2$ (Acmite) major Balance of iron oxide present largely as Fe_2O_3 & Fe_3O_4 Loose sludge on surface of scale largely analcite with some Fe_2O_3 & Fe_3O_4 also present The presence of $3Ca_3(Po_4)_2\cdot Ca(OH)_2$ (Hydroxyapatite) indicated only by faint appearance of the strongest line of its diffraction pattern
Laboratory Y	Surface of scale next to tube Cu_2O (Cuprite) major constituent CuO (Tenorite) minor constituent Powder scraped from surface of scale next to tube Mg_2SiO_4 (Forsterite) major constituent Cu_2O (Cuprite) minor constituent Composite of entire sample Mg_2SiO_4 (Forsterite) major constituent $CaCO_3$ (Calcite) minor constituent Powder scraped from surface of scale in contact with water Mg_2SiO_4 (Forsterite) major constituent $3MgO\cdot 2SiO_2\cdot 2H_2O$ (Serpentine) minor constituent	Surface of sample in contact with boiler tube Fe_2O_3 (Hematite) major constituent $Na_2O\cdot Al_2O_3\cdot 4SiO_2\cdot 2H_2O$ (Analcite) $Na_2O\cdot Fe_2O_3\cdot 4SiO_2$ (Acmite) very minor constituent Composite sample $Na_2O\cdot Al_2O_3\cdot 4SiO_2\cdot 2H_2O$ (Analcite) major constituent $Na_2O\cdot Fe_2O_3\cdot 4SiO_2$ (Acmite) considerable present Fe_2O_3 (Hematite) considerable present Surface of scale in contact with boiler water $Na_2O\cdot Al_2O_3\cdot 4SiO_2\cdot 2H_2O$ (Analcite) major constituent Fe_2O_3 (Hematite) Analcite was present in the largest particle size and acmite was the least perfectly crystallized.

Three laboratories submitted results of X-ray studies of the samples and these data are shown in Table V. It is interesting that agreement between laboratories is practically complete, not only on composite samples but on the various layers submitted to test. In the case of both deposits, the difference in composition of these layers is of considerable significance in interpreting mechanism of scale formation.

Positive identification of all major and most minor constituents has been obtained, and we are now able to formulate definite conclusions regarding the two samples. Before presenting these conclusions, however, let us bring the merits of the various types of examination into proper focus. It should not be inferred that the petrographic and X-ray methods are self sufficient. Each is a tool which provides supplemental information, and each has its own special field of application. The same may also be said of microchemical and spectrographic methods even though they are not discussed in detail in this symposium. We must rely on chemical analysis to tell us how much brick, how much wood, and how much cement we have to build with; the supplemental methods tell us whether we are building a church or a school and dictate the design.

To summarize briefly the data obtained from the three types of analysis we find that magnesium silicate, calcium carbonate, and copper oxide comprise about 95 per cent of the total of a composite of sample A, and that sodium aluminum silicate, sodium iron silicate, and iron oxides constitute approximately 83 per cent of the total of a composite of sample B. It would probably be unwise to attempt to specify exact percentages of each of these constituents as in so doing we must resort to hypothetical combinations to this extent: when one or more ions are common to two or more constituents, such as is the case with the analcite and acmite in sample B, the microscopist or X-ray technician must estimate the relative percentage of each constituent present as neither of these methods yields quantitative results.

The interpretation of the results of the various forms of examination that these samples have undergone, utilizing the specific and inferential information they reveal, has been so admirably accomplished by laboratory X that we quote directly from its report:

Interpretation—Sample A

"While calcium carbonate is ordinarily the predominant constituent of deposits found by heating hard bicarbonate waters, naturally soft or lime-soda softened waters frequently lay down magnesium silicate scales. This is particularly true where the water is heated to a temperature well above the normal level of 140 to 160 F.

"Magnesium silicate is consistently deposited from water as serpentine ($3MgO \cdot SiO_2 \cdot 2H_2O$) even at boiler temperatures, but is converted to forsterite (Mg_2SiO_4) on heating in the absence of water, especially at still higher levels. The change from forsterite in the layer adjacent to the tube wall to serpentine plus forsterite in the subsequently deposited layer is an indication of the overheating to which the heater coil was subjected as the scale progressively accumulated in it.

"The relative decrease in calcium carbonate or increase in magnesium silicate deposited during the latter part of the service life of this coil may possibly have been due to some change in the softening practice at the treating plant.

Interpretation—Sample B

"This rock-like deposit consisting essentially of sodium iron silicate, sodium aluminum silicate and iron oxide is an extreme example of what may happen when normal circulation in a boiler is upset by heavy overloading. While circulation apparently was adequate under normal operating conditions, it is probable that, at very high ratings, the water was nearly stagnant in the region where this deposit was formed.

"The mode of operation may be pictured as follows: Iron oxide formed elsewhere in the boiler and carried in suspension by the boiler water was dropped in the more or less stagnant water in the lower part of the upper-row tubes. At the same time this water was being concentrated, depositing analcite and developing a highly caustic solution containing much silica. The caustic solution acted to dissolve not only part of the iron oxide dropped from suspension but also some of the steel of the underlying tube metal, forming acmite interspersed with the analcite and residual iron oxide."

In conclusion, the committee wishes to express its deepest gratitude to the W. H. & L. D. Betz Co., the National Aluminate Co., the Cyrus William Rice Co., Hall Laboratories, Inc., and the Allis-Chalmers Manufacturing Co. These companies not only were generous of their time and talent in cooperating in the round-robin test program, but, fully realizing that they would to some extent be "put on the spot," they nevertheless consented to make this contribution towards the symposium.

[See General Discussion, p. 1308.—ED.]

X-RAY DIFFRACTION METHODS IN THE STUDY OF POWER PLANT DEPOSITS

By C. E. Imhoff[1] and L. A. Burkardt[1]

Synopsis

The procedure of the X-ray powder diffraction method in the study of power-plant deposits is briefly described. Characteristics of diffraction patterns and their interpretations are given. The diffraction analysis and chemical analysis of 22 deposits are compared to show fundamental differences between the two methods. The advantages and limitations of the diffraction method are discussed and patterns are shown which illustrate these specific points. In addition to compound identification, the method may give information concerning particle size, crystal modification, formation of solid solutions, and a rough quantitative estimation of the crystalline species present.

The problem of preventing the recurrence of undesirable power plant and water-formed deposits presents several questions which must eventually be answered before final conclusions are reached concerning preventative measures to be taken. These questions, in the order in which they may be asked, are: what is it; where was it found; under what conditions was it formed; and how can it be prevented?

The answer to the first question, which furnishes the initial step in the solution of the problem, depends upon the type of examination given the sample and who made the examination.

The methods of examination may be classified principally as chemical analyses and instrumental analyses. The chemical analysis involves the complete disintegration of the original sample by taking it into solution, and then removing from solution the various elements in such form that they may be weighed or measured. This analysis is then essentially an analysis of a solution containing the elements originally in the sample. Many attributes and properties of the deposit are not revealed by a quantitative determination of the chemical elements in this solution.

Instrumental analysis usually does not involve the disintegration or destruction of the original form of the sample but tests various of the physical properties which the material possesses. The chemical analysis must, therefore, be supplemented by instrumental analyses in order to get the most complete information possible. The purpose of this paper is to point out some of the things which an X-ray diffraction analysis is capable of revealing about deposits. To illustrate the differences between chemical analysis and X-ray diffraction analysis, let us suppose that the two methods of approach are applied to the examination of a rubbish heap. The reports, as shown in Table I, would be typical.

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The chemical analyst employing the general methods of analytical chemistry would first take apart all of the individual pieces, and this would correspond to taking a sample into solution. He would then pick out all of the wooden pieces, gather them together in one pile, and weigh them. This would correspond to the precipitation of a particular chemical compound and preparing it in such a condition as to be suitable for weighing. After the wood was separated would come the rubber, steel, and cotton in a certain definite order; and after the separations and weighings were completed, the findings would be reported in percentages of the total weight.

TABLE I.—ANALYSIS OF A RUBBISH HEAP, ACCORDING TO METHODS OF A CHEMICAL ANALYST AND AN X-RAY DIFFRACTIONIST.

Chemical Analyst		X-Ray Diffractionist
Wood	54.8 per cent	2 to 3 Wheelbarrows
Rubber	3.0 per cent	1 to 2 Bicycles
Steel	42.0 per cent	3 to 4 Rubber tires
Cotton	0.2 per cent	5 to 8 Torn shirts

The X-ray diffractionist, however, would not disturb the heap in any way, but would peer into the interior of the pile and recognize certain articles and would make note of the approximate number of these different articles which were observed. The wood, rubber, steel, and cotton would be reported in the form of wheelbarrows, bicycles, rubber tires, and torn shirts.

The chemist's report, in certain instances, would be entirely adequate, but it can be readily seen that the diffractionist's report adds much to the question of what materials are present in the rubbish heap.

In many ways, the chemical analysis of a power plant deposit can likewise be supplemented with considerable value by the report of an X-ray diffractionist. Just as in the above illustration, the diffraction analysis tells in what form the various constituents are combined, so also, in deposits, the diffraction analysis tells how the various chemical elements are combined.

EQUIPMENT

The equipment used in making X-ray diffraction patterns consists essentially of an X-ray tube, a source of high-voltage electrical current, a circular camera having a sample holder in the exact center, and a film scale. The tube is the heart of the unit in that it is the source of X-rays. High-voltage current is necessary for the production of X-rays, and this voltage should be adjustable and constant in order to control the intensity of the X-ray beam. The diffraction camera is circular so that diffracted rays may be intercepted by the photographic film regardless of the angle of diffraction. Figure 1 shows two such diffraction cameras equipped with small telechron motors for oscillation of the sample.

INTERPRETATION OF X-RAY PATTERNS

The X-rays are generated from the X-ray tube and passed through a collimator tube in order to produce a small beam of parallel X-rays. These are allowed to strike the sample, which is located in the exact center of the camera, and as these X-rays strike the various crystal planes, diffraction is produced at definite angles depending upon the distance between the ionic planes in the crystal.

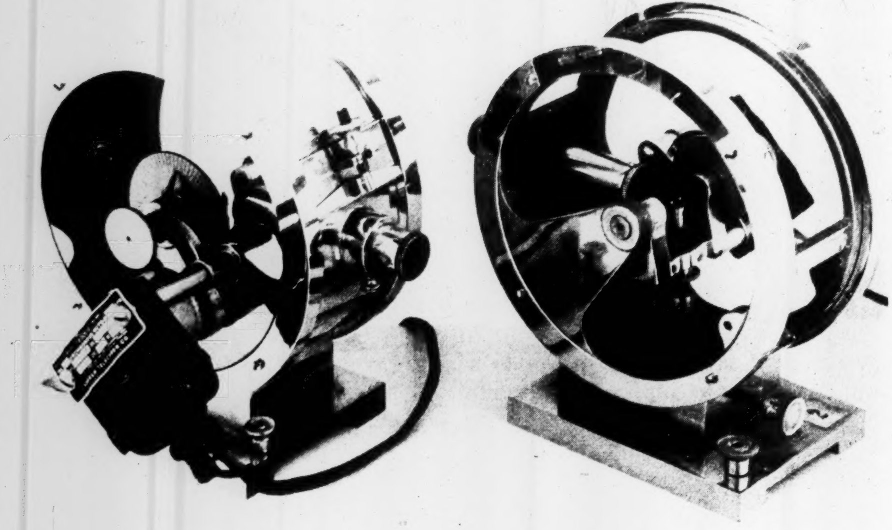

FIG. 1. Diffraction Cameras with Wedge Specimen Holders.

The two views of the diffraction cameras above show the collimator tube, the wedge-shaped sample holder, and motors for oscillating the sample. Oscillation gives smoother diffraction lines when the orientation of crystals in the sample is not completely random. The screw nut keeps the film holder tightly in place around the circumference of the camera.

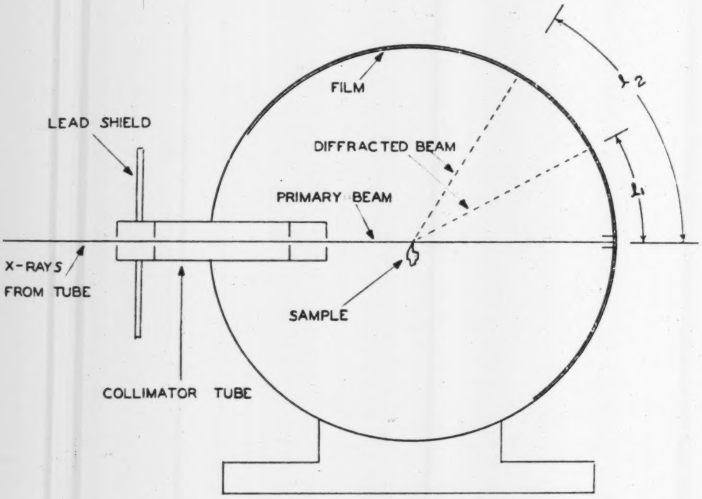

FIG. 2.—The Path of Diffracted X-rays.

X-rays enter the camera through the collimator tube and strike the sample at the center of the camera. Part of the beam is diffracted at angles depending upon the crystalline material in the sample. The diffracted beam strikes the photographic film which is at the circumference of the camera. Upon development of the film, there is produced a visible diffraction line. The length of the arc as measured between the diffraction line and the zero point (point of zero diffraction) is designated as **l**.

A sketch of the diffraction camera to show how the X-ray beam is diffracted from the main sample at various angles is shown in Fig. 2. The diffracted beams strike the X-ray film at various positions along the circumference of the camera. The distance between the diffracted beam and the undiffracted beam on the film is designated as l. These measurements are made as in Fig. 3. The length, l, is related to the distances between atoms or ions in the crystalline compounds in the sample, which distances are referred to as d values. The relationship between l and d is given by the formula

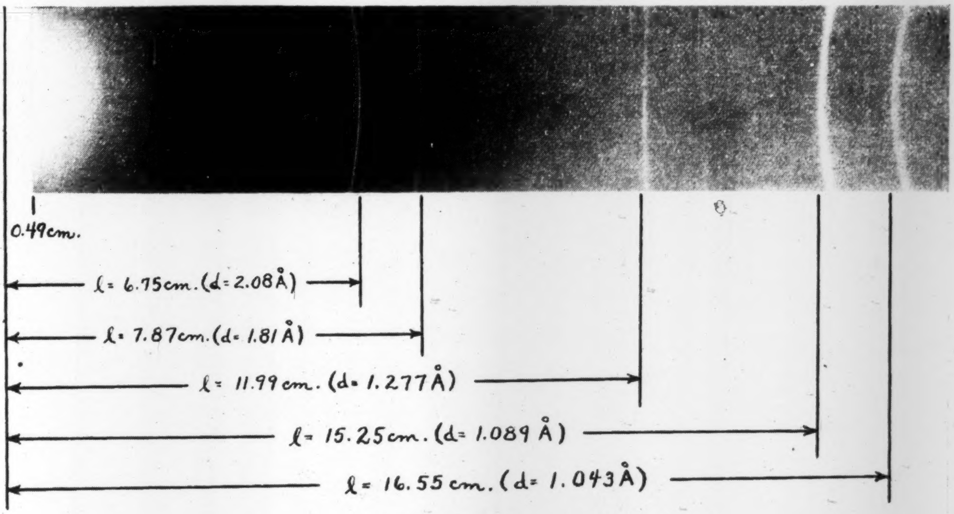

FIG. 3.—The Measurement of l Value.

The measurement of l is made on a developed film by means of a film scale. These measurements are made to the nearest hundredth of a centimeter. The true position of a diffraction line is taken to be at the middle of the line and at the middle of the film. The distance between the zero point and the diffraction line is the l value. From these the d values can be calculated.

$$d = \frac{n\lambda}{2 \sin\left(\frac{180\,l}{2\pi r}\right)}$$

where:

n = order of diffracted beam,
λ = wave length of the X-ray beam,
l = measured length on the X-ray film between the diffraction line and the zero point on the film,
r = radius of the camera, and
d = distance between parallel planes of atoms in the crystal lattice.

Referring to the above formula, the lines usually appearing on the pattern are of the first order of diffraction and n may be considered as one (1). Lambda (λ), the wave length, is constant for the X-ray tube and depends upon the atomic number of the metal used as the tube target. The radius, r, of the camera is also constant. The variables are, therefore, d and l. The l values are measurable, and from these the d values can be calculated.

In order to secure the maximum amount of information from an X-ray pattern, one should be able to recognize the characteristics of a pattern. Essentially what the X-ray does is to mark off on a circular film a series of linear dis-

TABLE II.—IDENTIFICATION OF A TURBINE BLADE DEPOSIT BY MEANS OF THE A.S.T.M. CARD INDEX OF X-RAY DIFFRACTION DATA.

Data from Diffraction Pattern			Data from Card Index	
l Values, cm.	Intensity	*d* Values, Å	*d* for Quartz, Å	*d* for Halite, Å
3.21	4	4.24	4.25	....
4.09	8	3.35	3.35	....
4.90	10	2.81	....	2.81
7.08	9	1.99	....	1.99
7.87	4	1.81	1.82	....
8.94	5	1.62	....	1.63
9.48	2	1.54	1.54	....
10.58	3	1.406	....	1.140
10.83	3	1.381		....
10.91	4	1.372	1.375	....
12.25	5	1.257	....	1.260
13.14	1	1.196	1.200	....
13.97	3	1.148	....	1.150

tances. It is as if the X-ray had inspected the sample with a micro-micrometer and had jotted down all the interplanar spacings it could measure within the various crystals. These are recorded in terms of linear distances (known as *l* values) on the film which are related to the measured interplanar distances (known as *d* values) according to the formula given above.

The diffraction lines are not all of equal intensity or quality, and there are times when a rather untidy pattern is produced with quite a dark background. When pinholes are used for collimation, the curvature of the lines changes from concave for the closer lines to convex for the farther lines. The true position from which *l* is measured is the exact center and middle of each line.

One method of identifying compounds consists of placing patterns of two samples side by side so that their zero points coincide. If there is a sufficient number of lines from one pattern matching those of another, and in the same orders of intensity, then that compound is present in both samples. For example, Fig. 4 shows a pattern of the mineral analcite compared to the pattern of an unknown sample of boiler deposit. It will be observed that the lines of the analcite mineral are duplicated in the unknown pattern, proving that analcite is a constituent in the deposit.

When a diffraction pattern is obtained for which no match can be found from the library of standard patterns, identification may be made by referring to data such as that contained in the A.S.T.M. Card Index of X-ray Diffraction Data.[2]

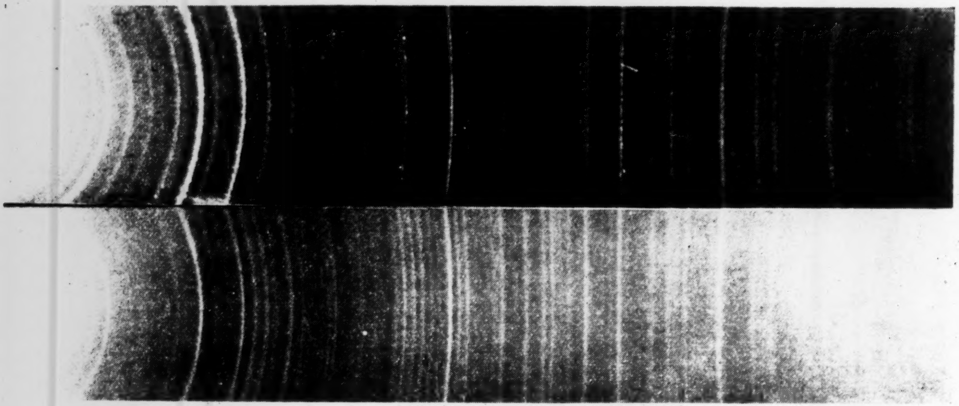

FIG. 4.—Identification by Visual Comparison.

The pattern of a scale sample (lower) is placed side by side with a pattern of the mineral analcite (upper). Identical positions of many of the lines in their proper orders of intensity identifies one of the constituents of the scale sample as analcite.

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[2] Card Index of X-ray Diffraction Data published jointly by the American Society for Testing Materials and the National Research Council (1942). (Issued as separate publication.)

This file is based on the work of Hanawalt, Rinn and Frevel.[3] The procedure is to first measure the l values of each diffraction line on the pattern and calculate the corresponding d values according to the formula previously given.

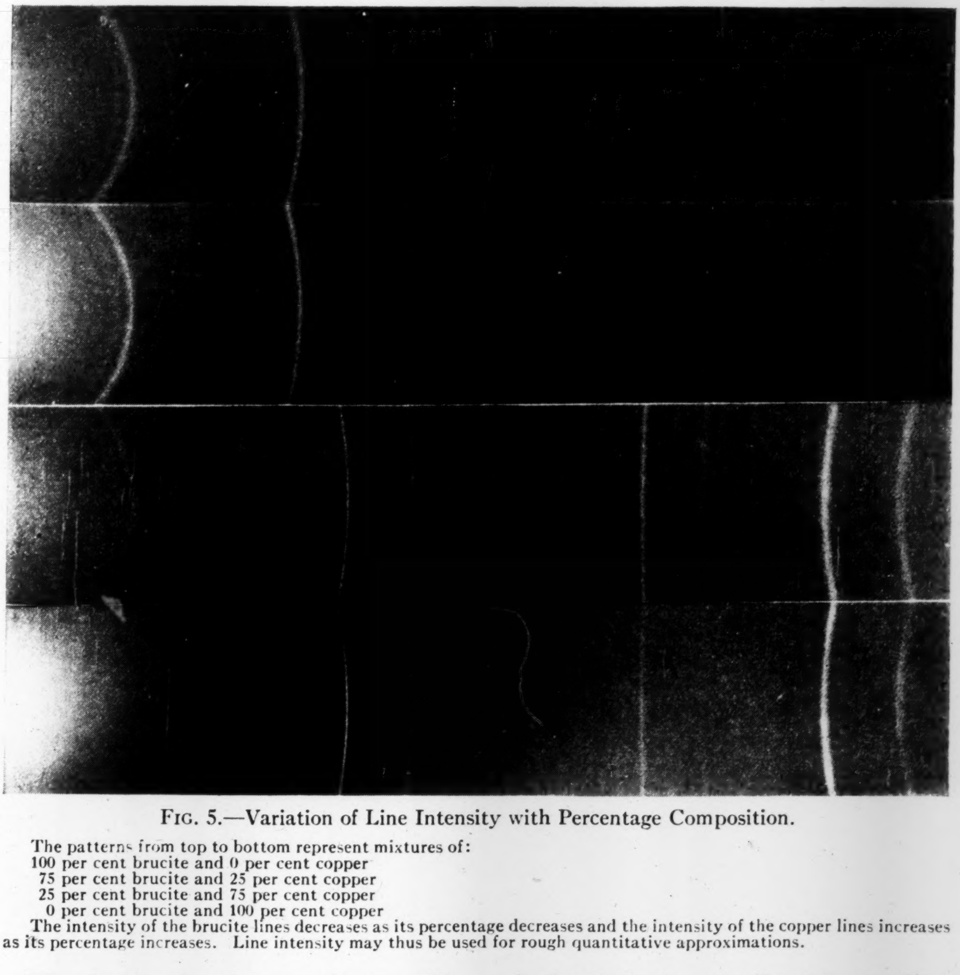

FIG. 5.—Variation of Line Intensity with Percentage Composition.

The patterns from top to bottom represent mixtures of:
100 per cent brucite and 0 per cent copper
75 per cent brucite and 25 per cent copper
25 per cent brucite and 75 per cent copper
0 per cent brucite and 100 per cent copper
The intensity of the brucite lines decreases as its percentage decreases and the intensity of the copper lines increases as its percentage increases. Line intensity may thus be used for rough quantitative approximations.

The d value for the most intense diffraction line is noted and the card index is consulted to see which compounds give this particular line as most intense in its diffraction pattern. For example, an identification of a turbine blade deposit is to be made from a pattern and no standards are available. The measured l values are listed in the first column of Table II. The corresponding calculated d values are listed in the third column. The units are Ångström units. The second column lists the estimated intensities based on the value of ten for the strongest line. The fourth and fifth columns contain data from the index cards for the compounds quartz and halite, respectively.

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[3] J. D. Hanawalt, H. O. Rinn and L. K. Frevel, "Chemical Analysis by X-Ray Diffraction," *Industrial Engineering Chemistry*, Analytical Edition, No. 10, pp. 457-512 (1938).

Continuing with the identification procedure, it is noted that the strongest line in the pattern of the blade deposit has a *d* value of 2.81 Å. Referring to the card index, it is found that there are 14 compounds including halite whose X-ray patterns have their strongest line at 2.81. Further examination shows that a number of the *d* values recorded on the card for halite are present in the pattern from the turbine blade deposit, and the relative orders of intensity are the same. There are, however, a number of lines which are not matched by the halite data, so the strongest of the remaining lines is selected, and the process of referring to the file is repeated. The most intense remaining line is the one with a *d* value of 3.35. There are 13 compounds in the card file with 3.35 as the strongest line, but the values for quartz are the only set which match the remaining lines for the blade deposit. Thus, the calculated *d* values of the unknown are all matched by recorded values of halite and quartz and the constituents of the blade deposit are identified as these two substances.

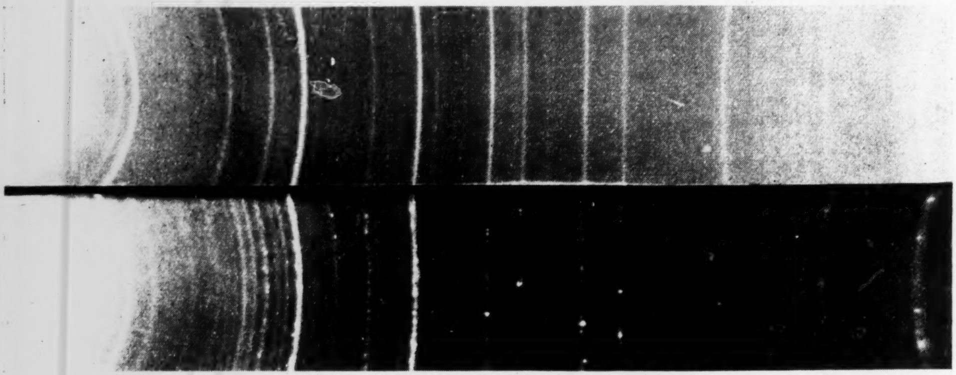

FIG. 6.—Line Texture and Particle Size.

Particle size and distribution may be indicated by the "texture" of the diffraction lines. The upper pattern of brucite shows by the uniform line density a uniformity of particle sizes of the individual crystals. The brucite lines in the lower pattern are slightly broader to indicate smaller crystals, but also there are some larger crystals present to cause speckles within the lines. In the latter sample there is a wide range of particle size distribution.

It is characteristic of X-ray patterns that the intensities of the lines vary considerably. The changes in intensity are due to different diffracting efficiencies of the atomic planes in the individual crystal species. However, the intensity also depends on the relative percentages of material present in a sample and upon the absorption of diffracted rays by other material.

Figure 5 shows how the intensities of the diffraction lines vary with percentage of composition in a mixture of brucite and copper. As the percentage of brucite decreases, the intensity of the brucite lines decreases and as the percentage of the copper increases the intensity of the copper lines increases. The relation between intensity and per cent is not absolute for one compound, however, but is influenced by the absorption characteristics the other compounds present. For this reason, the intensity of brucite lines from a mixture of 25 per cent brucite in copper is not the same as from a mixture of 25 per cent brucite in hydroxyapatite. In these two instances, copper absorbs a different amount of the diffracted rays from brucite than does hydroxyapatite.

Diffraction lines differ in breadth according to the grain size of the material in the sample. As a rule, the smaller the grain size, the greater is the breadth of the diffraction line. Particles of the order of 1000 Å give quite sharp lines, and as the particle size increases above this value, the lines become speckled. In Fig. 5, the lines for brucite are considerably broader than for copper, indicating a smaller particle size for brucite. An example of large particle size for brucite is shown in Fig. 6. The lower pattern in which the lines are speckled contained platelets of brucite which were visible to the unaided eye. The speckles are caused by the platelets. The upper pattern is one of brucite of normal and uniform particle size.

TABLE III.—CRYSTALLINE CONSTITUENTS OF POWER PLANT DEPOSITS IDENTIFIED BY X-RAY DIFFRACTION.

Name	Formula	Occurrence
Acmite	$Na_2O \cdot Fe_2O_3 \cdot 4SiO_2$	Boiler scale
Analcite	$Na_2O \cdot Al_2O_3 \cdot 4SiO_2 \cdot 2H_2O$	Boiler scale and sludge
Anhydrite	$CaSO_4$	Boiler scale
Aragonite	$\lambda CaCO_3$	Feedline and pumps
Bayerite	$\alpha Al_2O_3 \cdot 3H_2O$	Corrosion deposit
Bohmite	$\gamma Al_2O_3 \cdot H_2O$	Oil filter floc
Brucite	$Mg(OH)_2$	Boiler scales, softener sludges, feedline deposits
Bunsenite	NiO	Turbine blade corrosion
Burkeite	$Na_2CO_3 \cdot 2Na_2SO_4$	Turbine blade deposits
Calcite	$\beta CaCO_3$	Boiler scale and sludge, softener sludge, feedlines
Calcium Aluminate	$3CaO \cdot Al_2O_3 \cdot 6H_2O$	Softener chemical tank
Calcium Hydroxide	$Ca(OH)_2$	Boiler scale, superheater deposits
Cancrinite	$4Na_2O \cdot CaO \cdot 4Al_2O_3 \cdot 2CO_2$ $9SiO_2 \cdot 3H_2O$	Superheater deposits
Chalcocite	Cu_2S	Turbine blading
Cristobalite	SiO_2	Ignited turbine blade deposits
Cuprite	Cu_2O	Preheater corrosion product
Ferrous Oxide	FeO	Superheater, boiler tube
Forsterite	Mg_2SiO_4	Heat exchanger
Gypsum	$CaSO_4 \cdot 2H_2O$	Evaporator tubes
Halite	$NaCl$	Turbine blades
Hematite	Fe_2O_3	Boiler scale, corrosion deposits
Hemihydrate	$CaSO_4 \cdot O \cdot 5H_2O$	Boiler scale
Hydroxyapatite	$Ca_{10}(OH)_2(PO_4)_6$	Boiler scale and sludge
Magnetite	Fe_3O_4	Corrosion deposits
Metallic Copper	Cu	Boiler scale and sludge
Metallic Iron	Fe	Superheater corrosion
Metallic Silver	Ag	Dezincified silver solder
Natrolite	$Na_2Al_2Si_3O_{10} \cdot 2H_2O$	Boiler scale
Noselite	$Na_8Al_6Si_6O_{24}$	Boiler scale
Opal	SiO_2 (Amorphous)	Turbine blade deposit
Pectolite	$Na_2O \cdot 4CaO \cdot 6SiO_2 \cdot H_2O$	Boiler scale
Phosphorite	$Ca_3P_2O_8 \cdot H_2O$	Hot phosphate softener
Quartz	SiO_2	Boiler scale, economizer deposits, turbine blade deposits
Serpentine	$3MgO \cdot 2SiO_2 \cdot 2H_2O$	Boiler scale
Tenorite	CuO	Ejector trap corrosion deposits
Thenardite	Na_2SO_4V	Boiler scale, turbine deposits
Whitlockite	$\beta Ca_3P_2O_8$	Phosphate line at boiler drum
Wollastonite	$\beta CaSiO_3$	Ignited xonotlite boiler scales
Xonotlite	$5CaO\ 5SiO_2 \cdot H_2O$	Boiler scale

EXTENT AND LIMITATIONS

The X-ray diffraction method of analysis can only be used to the fullest advantage if the extent and limitations of the method are completely understood. Since identification is essentially a comparison procedure, it

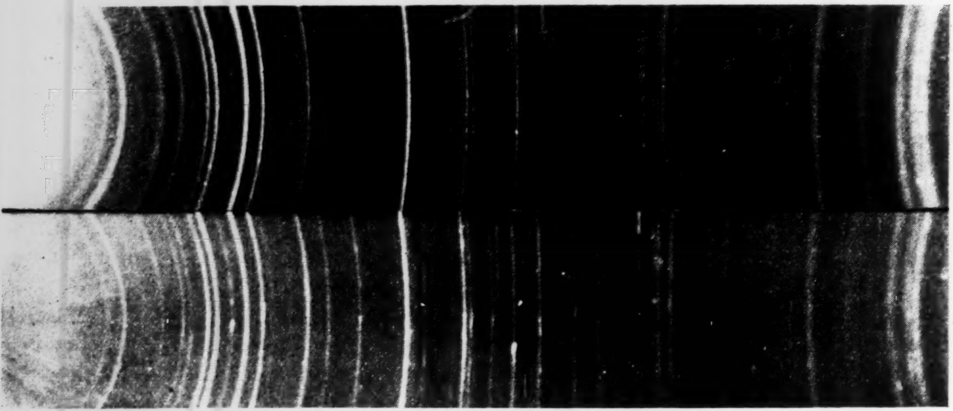

FIG. 7.—Solid Solution in a Dry Pipe.

The upper pattern is one of a dry pipe deposit; the lower of pure thenardite (sodium sulfate). The shifting of the diffraction lines in the pattern for the deposit, away from the positions for the lines in pure thenardite, indicates that thenardite in the deposit has taken into solution some constituent which has altered its lattice dimensions. Chemical analysis shows sodium carbonate to be the solute. The shifting of X-ray lines is also characteristic with solid solutions of metals (alloys).

TABLE IV.—CHEMICAL AND X-RAY ANALYSES OF TYPICAL POWER PLANT SCALES AND DEPOSITS.

Sample	Chemical Analysis, per cent												X-ray Identification
	SiO	R_2O_3	Fe_2O_3	CaO	MgO	SO_3	CO_2	P_2O_5	Cu	LOI	Na_2O	NaCl	
No. 1.—Scale from mud drum, 400-psi. steam pressure	13.6	36.0	...	6.7	3.0	...	...	5.7	1.2	25.4	...	...	hematite, graphite, apatite, magnetite
No. 2.—Scale from 400-psi. boiler middle drum	26.6	28.2	22.7	11.2	2.4	1.4	...	12.7	2.4	...	5.0	...	hematite, phosphorite, copper
No. 3.—Scale from 900-psi. boiler	19.7	...	26.6	10.8	16.2	0.1	...	12.3	...	8.5	...	...	hematite, serpentine, apatite
No. 4.—Scale from failed tube, 600-psi. steam pressure	10.9	...	18.5	19.[illegible]	9.2	...	...	28.1	...	1.7	...	...	apatite, magnetite, acmite
No. 5.—Scale from generating tubes, 400-psi. steam pressure	18.9	13.1	...	[illegible] 3	1.3	...	...	5.3	1.0	45.3	...	...	hematite, graphite, apatite, acmite
No. 6.—Deposit in phosphate line at entrance to boiler drum, 600-psi.	...	8.6	...	33.0	2.6	...	...	46.8		1.4	8.3	...	whitlockite
No. 7.—Boiler scale 1400-psi. steam pressure	32.2	47.0	...	4.7	0.1	0.2	...		5.4	1.2	...	...	acmite
No. 8.—Drum scale from rear drum of 650-psi. boiler	28.4	...	0.1	16.3	28.3	...	...	15.7	...	12.6	...	...	apatite, serpentine
No. 9.—Hot-process softener sludge	9.9	1.9	...	33.7	18.3	0.2	26.3	...	...	35.8	...	...	calcite
No. 10.—Waterwall header deposit from 250-psi. boiler	17.6	...	5.4	26.4	19.8	0.8	0.2	20.2	...	9.0	...	...	apatite, serpentine
No. 11.—Generating tube scale from 450-psi. boiler	40.4	45.8	...	20.1	4.4	...	...	3.5	...	5.3	...	...	xonotlite, hematite
No. 12.—Regulator valve deposit	10.4	5.3	...	7.1	48.7	0.1	...	...	...	27.9	...	...	brucite, calcite
No. 13.—Superheater deposit	7.8	...	3.0	35.2	0.7	9.4	...	...	0.8	9.1	...	11.1	calcium-hydroxide, halite
No. 14.—Sludge from 900-psi. boiler	24.8	...	5.7	16.4	22.8	0.3	...	7.7	...	8.8	...	...	analcite
No. 15.—Feed pump deposit	12.5	5.4	...	33.8	13.4	0.1	26.4	...	...	33.1	...	...	aragonite, calcite
No. 16.—Superheater deposit 400-psi. boiler	15.9	17.7	3.7	2.4	1.0	5.2	...	1.6	...	0.8	9.6	4.7	cancrinite
No. 17.—Superheater deposit 150-psi. boiler	1.4	0.6	...	0.9	...	29.0	16.8	...	...	...	48.3	0.7	burkeite
No. 18.—Dry pipe 150-psi. boiler	0.1	...	0.3	0.1	1.6	50.8	3.0	...	...	1.6	42.2	...	solid solution of Na_2CO_3 in thenardite
No. 19.—Sludge from waterwall tube of 1200-psi. boiler	0.6	...	44.0	25.4	2.8	0.7	...	24.8	...	2.2	...	...	hematite, magnetite, phosphorite
No. 20.—Deposit from superheater outlet 400-psi. boiler	61.3	...	16.7	6.2	0.7	7.5	...	0.5	...	7.4	...	...	quartz, hematite, anhydrite
No. 21.—Blade deposit from turbine using steam at 600 psi.	86.4	8.3	...	...	...	0.4	...	...	...	2.4	...	...	quartz
No. 22.—Economizer cyclone 1200-psi. boiler	0.3	...	9.8	41.5	3.1	16.3	...	27.5	...	1.2	...	...	apatite, magnetite

naturally follows that the only identifications which can be made are those making use of patterns of known compounds or of *d* values of known compounds.

Table III shows a list of compounds identified in power-plant deposits by the X-ray method. The table lists the crystalline constituents by their mineral names, the chemical formula, and the location in which they are found.

This list is not complete, for additional compounds will be identified as the various X-ray data on more compounds become available. In addition to the need for more data on new compounds, there is also the need for data on solid solutions. There is considerable evidence that the constituents of turbine-blade deposits may form a number of solid solutions. The authors have come to this conclusion after examining a number of diffraction patterns from turbine blading which, up to the present time, cannot be identified for lack of sufficient X-ray data and standard patterns. A solid solution may cause an alteration of the lattice of the solvent and thereby produce a shift in the position of the lines in the X-ray pattern. This is illustrated in Fig. 7, which shows a pattern for pure sodium sulfate compared with a pattern for a dry pipe deposit, consisting of a solid solution of sodium carbonate in sodium sulfate. It will be observed that there is a constant shifting of the X-ray lines as one proceeds from right to left across the pattern. The chemical analysis of this deposit is represented in sample No. 18 in Table IV.

The X-ray analysis can be used to some degree in making a quantitative estimation, provided the mixture is not a complex one. The method is not good for determining small amounts. Therefore, the failure of a particular compound to appear in a diffraction pattern means that it is not present in sufficient quantities to cause enough diffraction to activate the photographic film. The minimum percentages of material that can be detected in a sample may vary between 1 and 30 per cent, depending upon the atomic number of the elements in the compounds and upon the degree of crystallization. Table IV contains a list of representative scales and blade deposits with their chemical and X-ray analyses. It will be noted that the minimum percentage of material that can be identified varies over a wide range for different samples.

For example, sample No. 2 contained only 2.4 per cent metallic copper, but it was readily identified on the diffraction pattern. On the other hand, sample No. 14 contained 22.8 per cent of magnesium oxide and 16.4 per cent of calcium oxide, and yet no diffraction lines are observed for any of their compounds. Sample No. 22 showed 16.3 per cent sulfur trioxide, but no compounds of sulfur were found by the diffraction analysis. The usual assumption in such cases is that the combinations not visible in the diffraction pattern were present in an amorphous condition or were so poorly crystallized as to produce almost no diffraction.

Samples Nos. 1, 2, and 5 are from the same boiler but are from different locations. Although these samples have a number of similar chemical characteristics, acmite was formed in only one of them. Samples Nos. 3, 8, and 10 are typical of scales containing serpentine. This is usually associated with apatite or calcite. Sample No. 6 is unusual in that calcium phosphate has crystallized as whitlockite rather than apatite. Sample No. 7 gives a very pure pattern for acmite in spite of impurities indicated in the chemical analysis. Sample No. 9 is typical of all softener sludges in which calcite is the only compound sufficiently crystalline to produce diffraction. Magnesium and silica are amorphous. Sample No. 11 is xonotlite and hematite. Xonotlite is usually the form in which calcium silicate is found. Magnesium hydroxide will form large crystals if the growth is slow. Sample No. 12 represents a mixture of calcite and large crystals of brucite. Sample No. 13 is an unusual deposit consisting of calcium hydroxide and halite found in a superheater. Sample No. 15 is one in which the two crystalline forms of calcium carbonate—calcite and aragonite—were present. Sample No. 16 is a very complex deposit of cancrinite formed by the fusion of boiler water salts in a superheater. Sample No. 17 is also a superheater deposit but one in which sodium sulfate and sodium carbonate formed a double salt to be identical to the mineral burkeite. Sample No. 19 is a sludge from a high-pressure boiler in which the iron consists of both hematite and magnetite and the calcium phosphate is in a form identical to phosphorite. Sample No. 20 is a superheater deposit in which alteration has occurred to produce anhydrite as one of the crystalline species.

TABLE V.—LAYER ANALYSIS OF A SMALL PIECE OF BOILER SCALE.

Compound	Metal Side	Interior	Water Side
Quartz	X		
Magnetite		X	
Apatite	X	X	
Copper		X	
Unknown		X	X

SPECIAL TECHNIQUES

Ordinarily diffraction patterns are made from powdered samples ground to pass about No. 200 sieve. They may be mounted in the cameras in a variety of ways, such as in an oscillating wedge, in a revolving capillary, or on a piece of thread. Other methods of mounting the sample, however, may give information not possible with the ordinary powder procedure. Compounds in surface layers may be analyzed by mounting an unground sample so that the X-ray beam is intercepted by the surface at a very small angle. A small, thin piece of boiler scale of insufficient quantity for chemical analysis was mounted, as described above, to give a pattern of the surface of the scale adjacent to the boiler tube and again to give a pattern of the material in the surface in contact with the boiler water. A third pattern was made to give a pattern of the material in the main body of the sample. The resulting identifications are shown in Table V. The layer in the surface which was in contact with the boiler tube contained quartz and apatite. The interior of the scale contained magnetite, apatite, copper, and a material for which the standards necessary for identification were not available. The material in the surface of the scale contacting the boiler water consisted only of the unknown constituent identical to that in the sample from the interior.

This shows the nonhomogeneous nature of the small piece of scale which would certainly have escaped observation if a composite analysis were made.

The same technique may be applied to the analysis of surface layers on metal specimens. Small amounts of corrosion products can be analyzed by this procedure.

The most valuable information to be obtained from an X-ray analysis is that of positive identifications. In certain cases, however, negative identifications are of some value. It is sometimes well to know what a material is not. Negative identifications are frequently made on turbine blade deposits, particularly on those containing amorphous silica. When blank patterns are obtained on samples known to be very high in silica, the conclusion is that no crystalline material is present and, therefore, the silica is amorphous. If a sample of amorphous silica is ignited, crystallization usually proceeds with the formation of cristobalite or quartz depending somewhat on the temperature of ignition. Both of these crystalline compounds give good diffraction patterns.

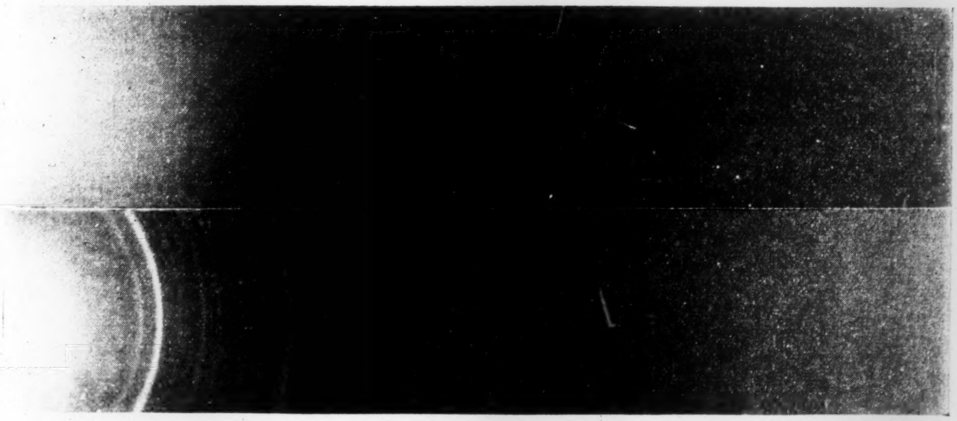

FIG. 8.—Crystallization of Amorphous Deposits.

The upper pattern having no diffraction lines indicates that the material in the blade deposit was amorphous. After igniting the sample at 1650 F. a pattern (lower) for cristobalite was obtained. The evidence from the two patterns is taken to indicate that amorphous silica was present in the original sample.

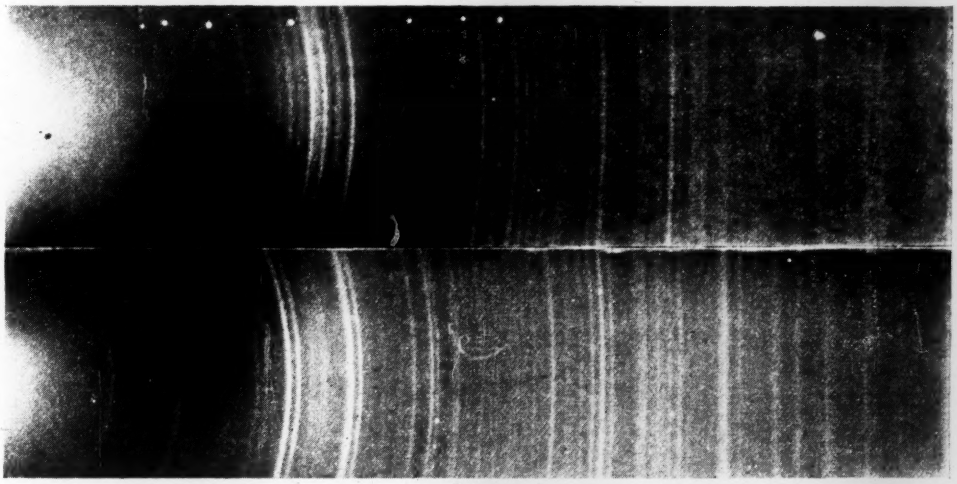

FIG. 9.—Increased Sensitivity by Acid Leaching.

The sensitivity of the X-ray method toward acid-insoluble compounds may be considerably increased by acid leaching. The upper pattern is from a boiler scale containing acmite in too small amounts to permit unquestionable identification. After leaching with acid a very strong pattern (lower) was obtained identical to acmite. Leaching had dissolved out the acid-soluble portion and left a residue mainly of acmite. Since the percentage of acmite was greatly increased, the intensity of the acmite pattern was greatly increased and simplification produced by eliminating lines from other compounds.

In Fig. 8 are shown patterns of an unignited and an ignited turbine blade deposit. The conclusion from the blank pattern is that no crystalline material was present and the ignited pattern shows cristobalite to indicate that crystalline silica is an ignition product. The conclusion is that free silica was present in the original sample and was in the amorphous form. This indirect method has its limitations, however, for certain compounds such as, for example, clay, may produce quartz or cristobalite on ignition.

Silicates are probably the most objectionable class of deposits encountered in the operation of boilers and turbines. The X-ray method may be made more sensitive in the identification of silicates by taking advantage of their acid insolubility. It is often possible to dissolve out of a sample all the acid-soluble materials and leave in the solid form only the insoluble silicates. Figure 9 shows a pattern of a scale from a high-pressure boiler and is represented by sample No. 4 in Table IV. The lower pattern is one made on an acid-treated sample. It is a very strong pattern for acmite. Although the acmite can be seen in the original pattern of the untreated sample, it is very weak and is practically invisible in the X-ray print. Acid treatment has converted the sample into one containing a very high percentage of acmite and as a result a very strong acmite pattern is obtained.

CONCLUSIONS

X-ray diffraction methods of analytical examination tell a great many things about a deposit which cannot be determined chemically. The most important attribute of the method is its ability to identify materials according to the chemical combination or crystal structure of the substance being examined. The X-ray analysis differs from the chemical analysis in that no alteration is given the sample and the record (patterns) is permanent. The interpretation of a pattern is not difficult, once the characteristics of patterns in general are understood. The X-ray method is very far from being a micromethod, but in certain samples surprisingly small percentages will produce a diffraction pattern. On the other hand, surprisingly large quantities have failed to give any diffraction patterns. This is usually interpreted as meaning that the material is amorphous. Special techniques, such as leaching and ignition, may be employed to produce a considerably greater sensitivity in the identification of the smaller percentages. Special mounting of the sample may make possible the analysis of small surface layers. These can be entirely different from the analysis of a ground-scale composite.

[See General Discussion, p. 1308.—ED.]

DIAGNOSIS OF WATER PROBLEMS AT LIMBO STATION

By Everett P. Partridge,[1] R. K. Scott,[1] and P. H. Morrison[1]

Hades, the after-world of the Greeks and Romans, was believed by them to lie in a subterranean region ruled by Pluto. As befits a locality from which few visitors are alleged to have returned, Hades and its borderland of Limbo have never become well known. Indeed, for thousands of years the most plausible description was that put into the mouth of Æneas by the Roman poet Vergil—to the discomfiture of successive generations of schoolboys.

Not until 1939 was there a report[2] on the problems of water conditioning involved in the operation of Limbo Station and its related industries. This great power plant might well be described as a mythical conglomeration of all boilers and related equipment that ever suffered what is called euphemistically "an unscheduled outage." At one end of the plant is the progenitor of the steam boiler and turbine, devised by Hero the Younger at Alexandria more than a century before the birth of Christ. It helps to carry the peak loads of wartime along with the latest high pressure giant at the other end.

The water problems of Hades and of Limbo are by no means limited to power generation. So many outmoded processes have been relegated to Limbo since the beginning of the industrial revolution that plants of all descriptions line the banks of the five great rivers. Each of these rivers, the sacred Styx, the placid Lethe, fiery Phlegethon, Acheron of the evil odors, and salt Cocytus, has its own combination of undesirable properties as a water supply. Moreover, the distribution system of the Metropolitan Water Commission of Hades is fed by wells whose waters are high in iron. Other well waters supplying various plants contain every imaginable constituent that ever plagued a plant engineer.

Merely as an indication of the stimulating variety of substances turned up in the run-of-mine flow of samples through the laboratories of the authors, Table I may be interesting. Emphasis rightfully must be placed, however, not on discovery but upon diagnosis. It is the intelligent engineering use of the fact that a given deposit contains acmite or calcite which counts, more than any amount of knowledge in an insulated vacuum.

The present series of items from Limbo is intended to stress the manner in which the diagnosis of water problems has been speeded and made more certain. When water produces an undesired result, it usually leaves some clue as to what happened. Most frequently these clues are in the form of materials which have been desposited from the water or produced by its action upon a confining surface. Chemical analysis gives much knowledge concerning the nature of such deposits or reaction products. Other techniques have been adapted, however, to aid the analytical chemist,

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[2] R. E. Hall, E. P. Partridge, and T. H. Daugherty, "Routing Nightmares of Water Conditioning," *Mechanical Engineering*, Vol. 10, pp. 715–721 (1939).

TABLE I.—SOME CONSTITUENTS OF DEPOSITS FROM WATER IDENTIFIED BY MEANS OF OPTICAL PROPERTIES AND X-RAY DIFFRACTION SPECTRA.

Chemical Name	Formula	Mineral Name	Occurrence in Industrial Water Systems
Aluminum			
silicate, sodium-	$3NaAlSiO_4 \cdot Na_2SO_4$	noselite	High-duty evaporative surfaces in boilers.
	$3NaAlSiO_4 \cdot Na_2S$	lazurite	Black liquor evaporators in Kraft paper mills.
	$Na_2O\ Al_2O_3\ 4SiO_2\ 2H_2O$	analcite	High-duty evaporative surfaces in boilers.
Ammonium			
bicarbonate	NH_4HCO_3	teschemacherite	Dead ends in lines carrying steam containing NH_3 and CO_2.
Barium			
carbonate	$BaCO_3$	witherite	Boiler supplied with water pretreated with $BaCO_3$.
sulfate	$BaSO_4$	barite	
Calcium			
carbonate	$CaCO_3$	aragonite	Heaters, condensers, heat exchangers.
	$CaCO_3$	calcite	Chemical-feed tanks and lines, lime-soda softeners, filters, distribution systems, heaters, boilers, condensers, heat exchangers.
-magnesium	$CaCO_3 \cdot MgCO_3$	dolomite	
fluoride	CaF_2	fluorite	Condenser (rare).
hydroxide	$Ca(OH)_2$	......	Evaporative surfaces in boiler, superheater.
oxalate	CaC_2O_4	......	Evaporators in beet-sugar plants.
phosphate	$Ca_3(PO_4)_2 \cdot H_2O$	......	Phosphate softeners, feed lines, economizers, boilers.
-hydroxide	$3Ca_3(PO_4)_2 \cdot Ca(OH)_2$	hydroxyapatite	Phosphate softeners, feed lines, economizers, boilers.
silicate	$5CaSiO_3 \cdot H_2O$	zonotlite	Evaporative surfaces in boilers.
sulfate	$CaSO_4 \cdot 2H_2O$	gypsum	Heat exchangers, condensers, process equipment, evaporators.
	$CaSO_4 \cdot 0.5H_2O$	......	Process equipment, evaporators.
	$CaSO_4$	anhydrite	Evaporative surfaces in boilers, process equipment.
-potassium	$K_2SO_4 \cdot CaSO_4 \cdot H_2O$	syngenite	Process lines and equipment (rare).
sulfite	$CaSO_3$	......	Chemical-feed tanks and lines, process equipment.
sulfide	CaS	oldhamite	Reduction product from $CaSO_4$ scale on overheated boiler tube.
Copper			
carbonate, basic	$CuCO_3 \cdot Cu(OH)_2$	malachite	Corrosion product on copper and brass tubing and equipment.
metal	Cu	......	Evaporative surfaces, drums and headers of boilers.
oxide, cupric	CuO	tenorite	Corrosion product on copper equipment.
cuprous	Cu_2O	cuprite	Evaporative surfaces, drums and headers of boilers, turbines, corrosion product on copper equipment.
Iron			
carbonate	$FeCO_3$	siderite	Steam lines.
oxide, ferric	Fe_2O_3	hematite	Evaporative surfaces, drums and headers of boilers.
hydrous	$Fe_2O_3 \cdot nH_2O$	goethite	Pumps, water lines, condensers, heat exchangers
magnetic	Fe_3O_4	magnetite	Heaters, economizers, boilers, superheaters, turbines.
silicate, sodium-	$Na_2O \cdot Fe_2O_3 \cdot 4SiO_2$	acmite	High-duty evaporative surfaces in boilers, superheaters.
sulfide	FeS	triolite	Superheaters.
Magnesium			
carbonate	$MgCO_3$	magnesite	Process lines and equipment.
-calcium	$CaCO_3 \cdot MgCO_3$	dolomite	Chemical-feed tanks and lines, lime-soda softeners, filters, distribution systems, heaters, boilers, condensers, heat exchangers.
hydroxide	$Mg(OH)_2$	brucite	Economizers, boilers.
phosphate	$Mg_3(PO_4)_2 \cdot (X)$	......	Economizers, boilers.
silicate	$3MgO \cdot 2SiO_2 \cdot 2H_2O$	serpentine	Heaters, heat exchangers, economizers, boilers.
	Mg_2SiO_4	forsterite	Overheated heater coils.
Potassium			
sulfate, -calcium	$K_2SO_4 \cdot CaSO_4 \cdot H_2O$	syngenite	Process lines and equipment (rare).
Silica			
crystalline	SiO_2	cristobalite	Boiler drum (rare).
	SiO_2	quartz	Turbines, evaporative surfaces in boilers.
amorphous	$SiO_2 \cdot nH_2O$	opal	Turbine (common), boilers, condensers (rare).

TABLE I.—*Continued.*

Chemical Name	Formula	Mineral Name	Occurrence in Industrial Water Systems
Sodium			
carbonate	Na_2CO_3	thermonatrite	Boiler tubes blocked by sludge, engine cooling jackets.
chloride	$NaCl$	halite	Superheaters, steam lines, turbines.
silicate			
-aluminum	$3NaAlSiO_4 \cdot Na_2SO_4$	noselite	High-duty evaporative surfaces in boilers.
-aluminum	$3NaAlSiO_4 \cdot Na_2S$	lazurite	Black-liquor evaporators in Kraft paper mills.
-aluminum	$Na_2O \cdot Al_2O_3 \cdot 4SiO_2 \cdot 2H_2O$	analcite	High-duty evaporative surfaces in boilers.
-iron	$Na_2O \cdot Fe_2O_3 \cdot 4SiO_2$	acmite	High-duty evaporative surfaces in boilers, superheaters.
sulfate	Na_2SO_4 V	thenardite	High-duty evaporative surfaces in boilers, superheaters, steam lines, turbines.
	Na_2SO_4 III	......	
Strontium			
sulfate	$SrSO_4$	celestite	Evaporative surfaces in boilers (rare).
Zinc			
carbonate	$ZnCO_3$	smithsonite	Corrosion product on galvanized pipe and equipment.
oxide	ZnO	zincite	Corrosion product from zinc strip in boiler
silicate	Zn_2SiO_4	willemite	Evaporative surfaces in boilers (rare).
sulfate	$ZnSO_4$	zinkosite	External deposits on leaking brass pipe.

or even, in some cases, to render his labors unnecessary. Of these, the identification of constituents by examination under the biological and the polarizing microscopes and by comparison of X-ray diffraction patterns will be illustrated by some examples. Although presented with less than the customary decorum, each of these is soundly based on the facts of actual cases.

A PIPE DREAM

"Hello, this is Pluto," said the resident manager into the telephone, which at once began to crackle. "What's that, the water main's corroded clear to Hades? Iron rust a half-inch thick in it? Probably have to replace most of it? Sure, I'll get someone to check into this!"

Thumbing through the list of eminent water chemists now residing in Limbo, he selected three names, called each man in turn, and arranged for each to inspect the water system on a different day. Then he called back to the engineer at the water plant, suggesting that what one investigator might say should not be communicated to the others.

Two weeks later, Pluto was reading their reports. Finishing the last, he carefully paraphrased their respective conclusions:

1. Professor A had been too busy to inspect the main himself. His assistant had, however, secured a sample of the material on the internal surface of the pipe. After precise chemical analysis he had found it to be substantially nothing but hydrous ferric oxide. Clearly, this was rust resulting from corrosion of the pipe by the water; a study should be made to determine how the water could be made less aggressive.
2. Dr. B had called in person at the water works in the Elysian Fields and had obtained a sample of water fresh from the wells. He had then traveled all the way down to the open-hearths in Tartarus and taken another sample. The analyses of these showed that the iron content decreased rather than increased as it passed through the line. The hydrous ferric oxide found on the pipe surface probably was deposited from the water rather than corroded from the

metal. Examination of a section removed from the line revealed no tubercles in the pipe, only a smooth gelatinous coating, with no pitting beneath it. The problem was that of preventing oxidation of the dissolved iron present in the water from the wells.

3. Mr. C had likewise checked the iron content of the water, but he had chanced to go down to the mills in Tartarus at a time when they were suffering from "red water." As a consequence, he found a large increase in total iron content over that in the well water. At first he, too, had suspected corrosion, but could find evidence of only the most superficial attack beneath the uniform deposit of gelatinous iron oxide. The possibility that dissolved iron present in the well water was being oxidized by air then was considered, but a careful examination of the system revealed no intentional aeration or probable air inleakage. Examination under the microscope did reveal, however, that the deposit was swarming with iron-fixing bacteria. Drastic treatment with chlorine to kill these organisms after stabilization of the dissolved iron at the well was recommended.

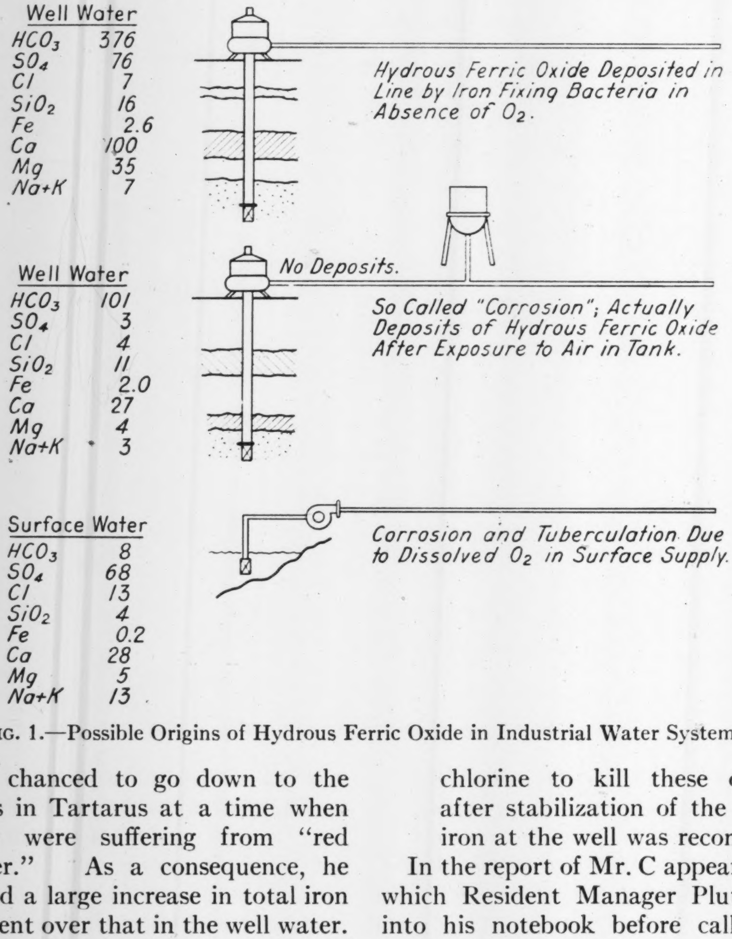

Well Water
HCO_3 376
SO_4 76
Cl 7
SiO_2 16
Fe 2.6
Ca 100
Mg 35
Na+K 7
Hydrous Ferric Oxide Deposited in Line by Iron Fixing Bacteria in Absence of O_2.
Well Water
HCO_3 101
SO_4 3
Cl 4
SiO_2 11
Fe 2.0
Ca 27
Mg 4
Na+K 3
No Deposits.
So Called "Corrosion"; Actually Deposits of Hydrous Ferric Oxide After Exposure to Air in Tank.
Surface Water
HCO_3 8
SO_4 68
Cl 13
SiO_2 4
Fe 0.2
Ca 28
Mg 5
Na+K 13
Corrosion and Tuberculation Due to Dissolved O_2 in Surface Supply.

FIG. 1.—Possible Origins of Hydrous Ferric Oxide in Industrial Water Systems.

In the report of Mr. C appeared Fig. 1, which Resident Manager Pluto copied into his notebook before calling in a consultant from the upper world to review the assembled information. When the survey by the latter supported Mr. C's conclusion that the "corrosion" was actually deposition of hydrous ferric oxide by iron-fixing bacteria, Pluto phoned his purchasing agent to get him figures on stabilizing chemicals and chlorination equipment.

"And we won't need that pipe I told you to get busy and order a month ago," he added before he hung up.

TABLE II.—IDENTIFICATION OF ACMITE PRODUCED BY ATTACK OF CONCENTRATED BOILER WATER ON CEILING OF BOILER TUBE.

		Per cent	Equivalents
Chemical analysis			
Sulfur trioxide	(SO_3)	0.5	0.013
Carbon dioxide	(CO_2)	trace	.....
Phosphorus pentoxide	(P_2O_5)	1.2	0.051
Silica	(SiO_2)	42.0	1.400
Iron oxide	(Fe_2O_3)	32.4	1.213
Aluminum oxide	(Al_2O_3)	0.8	0.047
Calcium oxide	(CaO)	2.3	0.082
Magnesium oxide	(MgO)	1.3	0.065
Sodium oxide	(Na_2O)	9.5	0.306
Copper	(Cu)	6.4	.....
Net ignition loss		3.5	.....
Identified constituents			
Sodium iron silicate $Na_2O \cdot Fe_2O_3 \cdot 4SiO_2$		74.6	.....
Copper Cu		6.4	.....
Iron oxide Fe_3O_4		5.6	.....
Probable minor constituents			
Sodium aluminum silicate $Na_2O \cdot Al_2O_3 \cdot 4SiO_2 \cdot 2H_2O$		3.4	.....
Calcium phosphate $3Ca_3(PO_4)_2 \cdot Ca(OH)_2$		2.8	.....
Magnesium silicate $3MgO \cdot 2SiO_2 \cdot 2H_2O$		3.0	.....

An Expensive Synthesis of Acmite

"That's the hardest deposit I ever tried to turbine out of a tube," said the assistant engineer as Pluto came past the uptake header end of the 1400-lb. cross-drum boiler. "The cutter damn near tries to chew its way through the bottom of the tube instead of chipping out the scale."

"Whaddya mean, bottom of the tube," replied Pluto, "isn't the scale on the bottom?"

"No, there's patches of it along the tops of the tubes in the two upper rows back a little ways from the uptake end," said the assistant.

"Send a sample in for analysis and let's find out what goes on," and the chief was on his way.

When the sample was analyzed, the service engineer for the consulting laboratory shook his head, for this, after all, was in 1938. "Is this any kind of a bunch of hash for you to turn out?" he remarked to the head of the laboratory, pointing to the chemical analysis in Table II. "Where's the aluminum oxide to go with the sodium oxide and silica to make analcite?"

"O. K., brother, but it isn't analcite, at least not much of it. I figured you'd be in, so I gave it to the girl on the microscope. I just got this report: 'A well-crystallized deposit of sodium-iron silicate, with considerable metallic copper and a small amount of sodium-aluminum silicate. The sodium-iron silicate is identical with the mineral acmite, $Na_2O \cdot Fe_2O_3 \cdot 4SiO_2$.'"

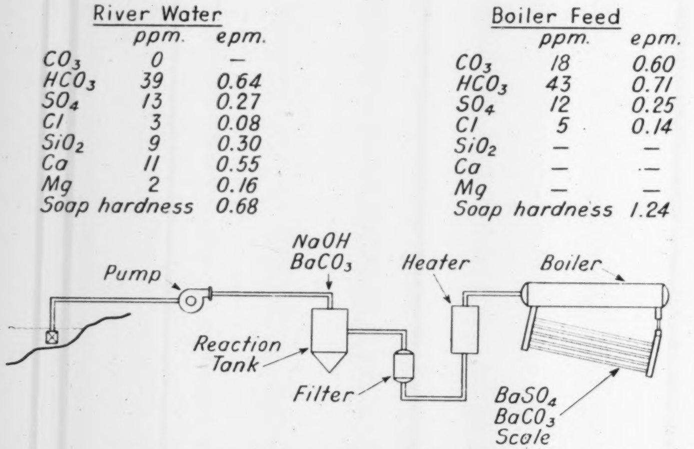
A quick determination of its X-ray diffraction pattern verified the fact that the substance was acmite.

Interpretation of the case was facilitated by the observation at the plant that the patches of sodium-iron silicate were associated with actual internal thinning of the tubes along the top. Obviously, the iron in the complex silicate was being supplied by the metal of the tube. The propensity of straight-tube cross-drum boilers for developing steam-blanketing at the upper ends of the upper rows had been recognized even before 1938. When it was learned that the boiler had recently been operated with increased caustic soda in the boiler water in an effort to lessen tube losses mistakenly attributed to ordinary corrosion, the picture was complete. Even though the body of the boiler water contained only 150 ppm. of NaOH and 40 ppm. of SiO_2, extreme concentration of this along the ceilings of the upper-row tubes had actually produced a solution capable of dissolving iron. From this solution the acmite had then crystallized.

During subsequent operation with low, controlled alkalinity the attack was so limited that further more elaborate steps were deemed unnecessary.

COLLECTOR'S ITEMS

Although calcium-sulfate boiler scale had almost the status of a regular by-product of power generation a quarter of a century ago, even the smaller industrial plants in Limbo rarely send in a sample today. Indeed, the presence of even a minor amount of anhydrite in a boiler deposit is regarded as a warning that adequate phosphate ion has not been present in the boiler water at the point where the crystals formed. Perhaps calcium sulfate will soon be as rare a constituent of boiler deposits as the unusual substances which provided the following interesting experiences:



River Water

	ppm.	epm.
CO_3	0	—
HCO_3	39	0.64
SO_4	13	0.27
Cl	3	0.08
SiO_2	9	0.30
Ca	11	0.55
Mg	2	0.16
Soap hardness		0.68

Boiler Feed

	ppm.	epm.
CO_3	18	0.60
HCO_3	43	0.71
SO_4	12	0.25
Cl	5	0.14
SiO_2	—	—
Ca	—	—
Mg	—	—
Soap hardness		1.24

FIG. 2.—Diagram of System Which Produced Barium Sulfate Scale in Boiler.

The analyst dropped the sample envelope on the microscope desk. "Here's a funny one for you," he said. "More than 50 per cent insoluble on digestion with HCl, but it doesn't look like any silica scale I ever saw before, and hydrofluoric acid doesn't touch it."

Nor did it look like a silica scale to the microscopist. All the particles were birefringent in polarized light—hence no amorphous silica, cristobalite, or sodium-aluminum silicate. The upper indices were far above those of quartz or the probable calcium or magnesium silicates. The microscopist settled down to the routine of measurement in one index liquid after another. Two major constituents were found—one much more birefringent than the other, with maximum and minimum indices of about 1.675 and 1.530—values of about 1.650 and 1.635 for the other . . . . .

The telephone rang in the control laboratory. To the analyst's ear came the voice of the microscopist—a voice with overtones of restrained triumph. "You know that scale with a lot of acid-insoluble material in it?—well, it isn't silica, it's barite—barium sulfate to you. And along with it there's quite a bit of witherite—barium carbonate."

Said the analyst, "You're nuts, who ever heard of a barium sulfate boiler scale?" But when his flame test showed green he reported the whole thing to the service engineer. "Well, that's not so screwy as it sounds," said the latter, "one of the few barium treatment units in the country is at that plant."

A check on the water entering the treating unit and leaving the filter showed that the hardness of the boiler feedwater was being nearly doubled by the barium entering solution, as indicated in Fig. 2. In the attempt to prevent calcium sulfate boiler scale by precipitating sulfate ion from the water before it entered the boiler, the formation of barium sulfate scale had actually been promoted. Before long the plant installed a lime-soda softener, followed by internal phosphate conditioning.

Three years later another microscopist and another analyst in the laboratory collaborated to round out the alkaline-earth sulfate scales. The first report from the microscopist read, "Very unusual sample whose main constituent is an acid-insoluble low-birefringent crystalline material with a refractive index of about 1.620." The analyst observed a red color in the flame test, separated calcium, still "saw red," and concluded that a strontium compound was present. With this knowledge, the microscopist identified the chief constituent as celestite, strontium sulfate.

A check on the make-up water from the deep well supplying the plant showed 24 ppm. of strontium ion. While the hot-process lime-soda softener removed much of the 65 ppm. of calcium ion also present in the water, much of the strontium escaped precipitation because of the greater solubility of strontium carbonate. No further treatment had been thought necessary. Adoption of internal conditioning with phosphate provided the answer to this unusual case by precipitating strontium phosphate along with calcium phosphate as sludge within the boiler.

With the roll-call of the alkaline-earth sulfates completed, the boy who checks in the samples is willing to bet that any day now a deposit of magnesium sulfate will turn up.

THE DEPOSIT OF CALCIUM (—?—)

One day a succession of mishaps at Limbo Station threw an unexpected sudden demand for power on a smaller plant on the river Phlegethon. As the output climbed to capacity and a bit beyond, the vacuum on the condensers dropped like a barometer at the approach of a tornado. The chief engineer sweated it out until the emergency was past, then, at the first opportunity, inspected his condensers. Finding the tubes covered with a rather soft, but compact, deposit, he called in a consultant, who procured samples of the deposit and of the cooling water from the river.

During the next few days, as the analyst in the consultant's laboratory worked on the samples of deposit, his customary confidence gave way gradually to consternation. There was practically no water-soluble material, the ignition loss was low, silica was negligible, calcium oxide was high, yet the sum of all items ordinarily determined was only a little over 75 per cent of the total.

Under the polarizing microscope, the sample appeared as finely divided isotropic particles with an unusually low refractive index, apparently coated with hydrous ferric oxide. Said the microscopist, "The only calcium compound I know of with a refractive index anywhere near as low as this is calcium fluoride." "Maybe you have something there," replied the analyst. And she had, for a special determination of fluorine closed the analysis.

A check on the water used as make up for the spray pond showed 45 ppm. of fluoride ion, together with 34 ppm. of calcium ion, in the river Phlegethon. Search for the source led to a glass plant a mile upstream which was discharging waste fluoride etching solutions into the river. Until something could be done about this, the power plant switched to well water for its cooling system.

THE MYSTERY OF THE DISAPPEARING DEPOSIT

On the east side of the black and malodorous river Acheron lies the starch plant of the lower regions, established by a group of enterprising executives in the hope of shortening their 100-yr. detention in Limbo before admission to the Elysian fields.

Among the major difficulties of starting a new plant, one minor incident caused much amazement and amusement. The first time the valve in the steam line to the driers was opened, no steam came through. After checking back and forth along the line, which had been unused for some months after the boiler plant went into service, the valve was removed and the difficulty was revealed as a solid plug of deposit just ahead of it in the line.

"It's obviously carry-over from the boilers," said the plant chemist, but after struggling for several hours over a semi-quantitative analysis, he was ready to tear out his few residual hairs. "What goes on here?" he complained to his assistant; "It's completely water soluble; it gives off as much carbon dioxide as a club soda, and yet there's no sodium!"

Late in the afternoon, he dumped a little of the deposit on a watch glass and took it over to the microscope laboratory. "See what you can make out of this, will you?" he requested. "Can't tackle it now," the microscopist replied, "but I'll look at it first thing in the morning."

Next morning the microscopist dropped into the chemist's office. "Say, if you wanted that sample back, why did you leave the watch glass behind."

"I didn't take it—the cleaning woman must have spilled the stuff."

"No, I had it in the cabinet alongside my oven. What in the name of Hecate did happen to it?"

"Never mind, I have plenty more here—why, some of this is gone, too!"

With the remainder of the sample the two dashed for the microscope. The crystals proved to be relatively large, well-formed, with parallel extinction and indices from a maximum of about 1.555 to about 1.425. As he looked through his tables the microscopist began to chuckle. He reached for a bottle of sodium hydroxide, ran a couple of drops onto a little of the sample on the slide and held it under his friend's nose.

"Ammonia! But what ——"

"It's ammonium bicarbonate. I'll leave it to you to figure out how it was laid down in that dead end."

Analysis of a sample of condensate from the power plant showed 4 ppm. of ammonia, while the bicarbonate waters of Acheron were found to contain 8 ppm. of free and 1 ppm. of albuminoid ammonia. The advisability of ammonia removal to protect copper-alloy equipment was obvious. Installation of a good deaerator following the lime-soda softener satisfactorily completed the cycle of events started by the disappearing deposit from the dead end of the steam line.

A HANDFUL OF CRYSTAL NUCLEI

Cooling water for the condensers in Limbo Station was originally used on a once-through basis from the river Styx. Then, in an effort to obtain lower temperatures, a recirculating system was installed with the most modern of cooling towers. Soon after, trouble with calcium carbonate scale led to the introduction of threshold treatment.

Only a few months later the heat transfer on No. 736 turbine condenser—Limbo Station has thousands of units—began to drop. When the condenser was opened, a powdery deposit was found in the tubes. Qualitative tests for phosphate, carbonate, and calcium were strong, leading to the conclusion that both calcium phosphate and calcium carbonate had been precipitated. Almost perfunctorily, the plant chemist shipped off the sample to the consultants.

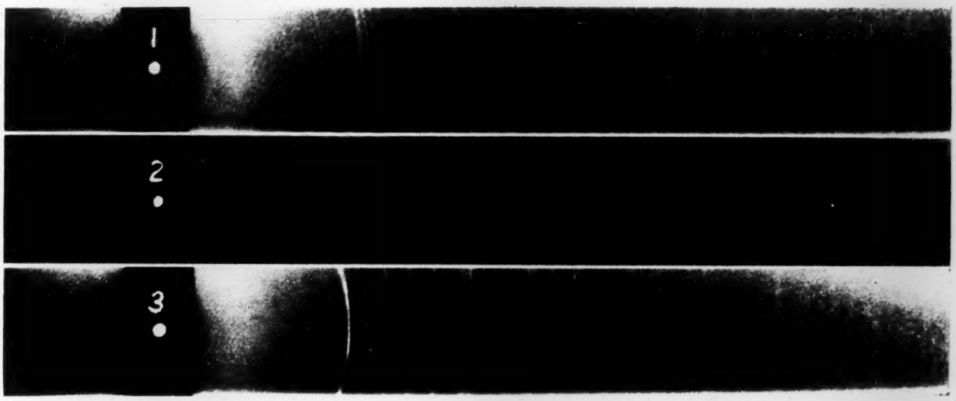


FIG. 3.—X-ray Identification Fails in the Case of an Amorphous Deposit.

1. Hydroxyapatite, $3Ca_3(PO_4)_2 \cdot Ca(OH)_2$.
2. "A handful of crystal nuclei," amorphous deposit with analysis corresponding to $3CaO \cdot 1P_2O_5 \cdot 1CO_2$.
3. Calcite, $CaCO_3$.

A preliminary microscopic examination failed to reveal crystals of calcium carbonate mixed with finely-divided calcium phosphate. As a matter of fact, none of the constituents could be resolved at 450 diameters. When chemical analysis revealed a molar ratio close to $3CaO:1P_2O_5:1CO_2$, debate ranged between the hypotheses that the sample was (1) a mixture of calcium pyrophosphate and calcium carbonate, both in submicroscopic particles, and (2) a complex carbonate-phosphate, perhaps similar to dahllite, recorded by the mineralogists as $7CaO \cdot 2P_2O_5 \cdot 1CO_2$.

The first hypothesis was eliminated when it was determined that practically all the phosphate was present in the ortho form, rather than as a molecularly dehydrated phosphate. To check the second hypothesis, the sample was subjected to the crucial test of the X-ray—with the mirth-provoking results evident in Fig. 3. Instead of the characteristic pattern of the calcium phosphates of the apatite group, the negative was innocent of any trace of a diffraction pattern. Although he swore that he had made no mistake, the X-ray specialist repeated his work on a new specimen, again drawing a blank. Here was something so devoid of crystal structure that it was amorphous not only to the microscope, but also to the X-ray.

Since any ordered arrangement of atoms in the material must have extended for less than about 100 Ångstrom units, the sample came to be known as "the handful of crystal nuclei." Further deposition of "nuclei" was eliminated by increased blowdown from the cooling system but the arguments among the technical staff have not yet abated.

THE ISOTROPIC RIDDLES

The microscopist came into the X-ray laboratory bearing a puzzled frown and

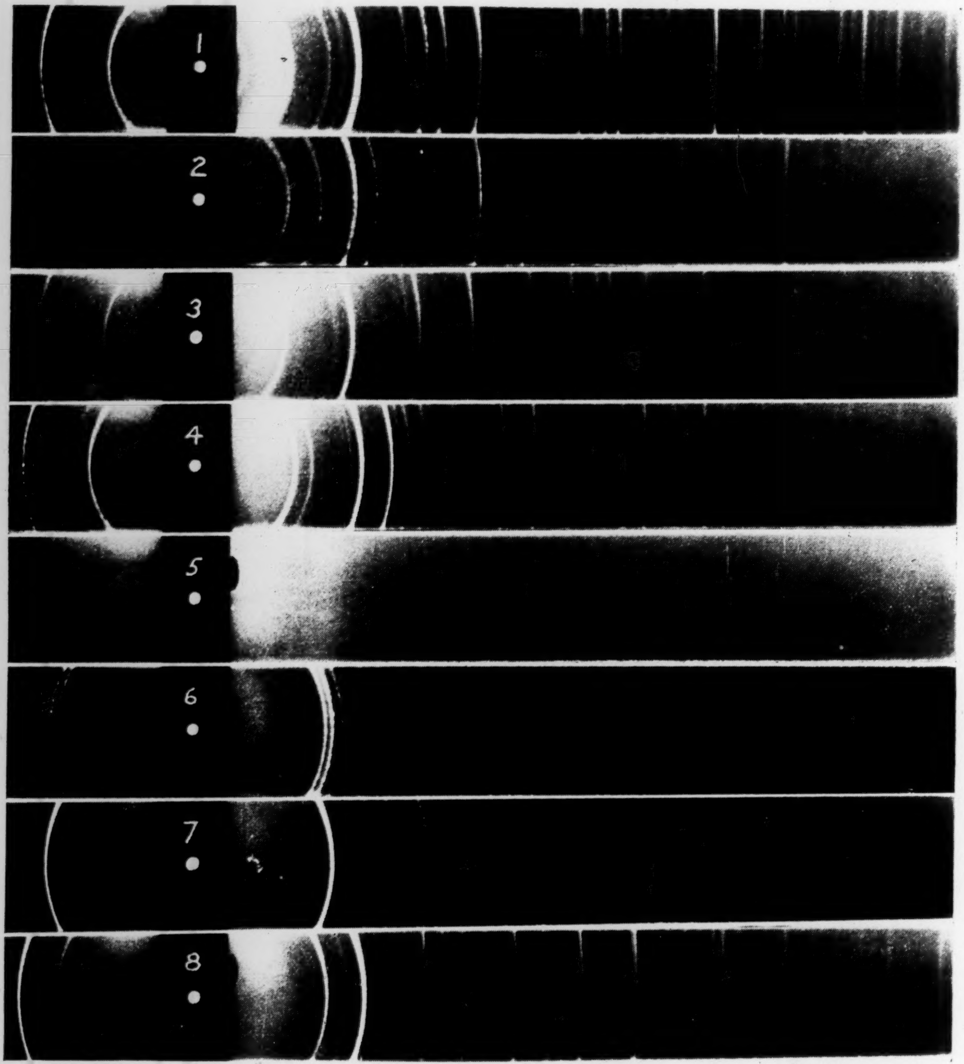


FIG. 4.—X-ray Examination Differentiates Between Silicate and Silica Deposits Difficult to Differentiate Under the Polarizing Microscope.

1. Sodalite mineral, $3NaAlSiO_4 \cdot NaCl$.
2. Deposit on top-row generating tubes, 1400 psi. Composition approximating mineral noselite, $3NaAlSiO_4 \cdot Na_2SO_4$.
3. Scale from black-liquor evaporator in sulfate pulp mill. Composition approximating mineral lazurite, $3NaAlSiO_4 \cdot Na_2S$.
4. Analcite deposit from high-pressure boiler, $NaAl(SiO_3)_2 \cdot H_2O$.
5. Amorphous silica from last stages of turbine, $SiO_2 \cdot nH_2O$.
6. α-Tridymite (SiO_2) found in chemical dissolving tank, probably small pieces of silica refractory, contains some α-cristobalite.
7. α-Cristobalite (SiO_2) separated from deposit under feed pipe in steam drum of boiler.
8. α-Quartz (SiO_2) from middle stages of turbine.

a handful of sample envelopes. "I'm stuck," she said. "Here's a consignment of deposits from all over Limbo; the major constituent of every blessed one except the last is isotropic with an index close to 1.485; the last one is quartz; they all contain quite a bit of silica, but the laboratory wants a clue as to their probable identity before they go on with the analysis."

The X-ray specialist read the descriptions—deposit on top-row generating tubes in No. 6 1400-psi. boiler—scale from black-liquor evaporator in sulfate pulp mill—hard scale from high-pressure boiler—deposit from last stages of turbine—insoluble lumps from chemical-dissolving tank—deposit from under feed pipe in steam drum. "They certainly are various," he commented; "suppose I run them off to see what turns up."

When he examined his negatives, they also looked various. It finally took complete analyses to unravel some of them but final conclusions were those expressed in Fig. 4. By comparison with a pedigreed sample of the mineral sodalite $3NaAlSiO_4 \cdot NaCl$, (1), it seemed probable that the deposit from the top-row generating tubes of the 1400-psi. boiler with an analysis approximating that of noselite, $3NaAlSiO_4 \cdot Na_2SO_4$, (2) was actually that member of the sodalite group of minerals. The scale from the black-liquor evaporator (3) which approximated in composition another member of the sodalite group, lazurite, $3NaAlSiO_4 \cdot Na_2S$, was tentatively identified as that substance. The analcite deposit (4) was an old friend, seen frequently before.

When the deposit from the last stages of the turbine (5) proved to be almost entirely silica and gave no diffraction pattern, it was obviously amorphous silica. The lumps from the chemical dissolving tank (6) and the deposit from under the feed pipe in the boiler drum (7) were also nothing but silica, but the X-ray was able to identify them respectively as α-tridymite and α-cristobalite. Just to complete the series for the forms of silica, the deposit from the middle stages of a turbine (8) was quartz.

FIG. 5.—X-ray Pattern of Magnesium Silicate of the Serpentine Type as Found in Deposits from Water.

Broad, diffuse bands indicating extremely small particle sizes are apt to be overlooked in presence of sharper lines of better-crystallized substances when these are present with magnesium silicate.

COOPERATIVE COMMON SENSE

Any inference from the preceding examples that the analytical laboratory may be closed up, once the necessary investment has been made in microscopic and X-ray equipment, is dead wrong. Chemical analysis still remains the reliable means of determining *how much* of various constituents a sample contains, while the other techniques are establishing *which* substances are actually present. A good example is afforded by the almost omnipresent magnesium silicate, which appears amorphous to the microscope and so nearly amorphous to the X-ray that it gives the characteristically diffuse diffraction pattern shown in Fig. 5. When admixed with calcium phosphate in boiler sludges or with calcium carbonate in heater deposits it is likely to be underestimated or missed entirely unless the analyst contributes his determinations.

TABLE III.—CHEMICAL ANALYSIS OF, AND MICROSCOPIC AND X-RAY IDENTIFICATION OF CONSTITUENTS IN A PHOSPHATE BOILER SLUDGE.

Microscopic Examination:
The major constituents of this sample appear to be magnetic iron oxide (magnetite) and finely-divided calcium phosphate (hydroxyapatite). Minor amounts of well-crystallized magnesium phosphate and of metallic copper are present. A chemical test for sodium water-soluble salts was obtained.

X-ray Identification:
The sample contains magnetic iron oxide (magnetite), calcium phosphate (hydroxyapatite) and magnesium phosphate as its chief constituents. Metallic copper is also present.

		Per cent	Equivalents
Chemical analysis			
Sulfur trioxide	(SO_3)	2.1	0.052
Carbon dioxide	(CO_2)	0.0	.....
Phosphorus pentoxide	(P_2O_5)	28.4	1.198
Silica	(SiO_2)	0.3	0.010
Iron oxide	(Fe_3O_4)	29.2	.....
Aluminum oxide	(Al_2O_3)	1.4	.....
Calcium oxide	(CaO)	11.6	0.414
Magnesium oxide	(MgO)	17.9	0.888
Copper	(Cu)	6.6	.....
Net ignition loss		2.1	.....
Identified constituents			
Magnesium phosphate $Mg_3(PO_4)_2$		36.2	.....
Calcium phosphate $3Ca_3(PO_4)_2 \cdot Ca(OH)_2$		20.4	.....
Magnetic iron oxide Fe_3O_4		29.2	.....
Metallic copper Cu		6.6	.....

Note.—Magnesium orthophosphate, actually the predominant constituent in this sample, was present only partially as crystals large enough to be identified under the microscope. Much of the magnesium phosphate indicated by chemical analysis must have been present, admixed with the calcium phosphate in the mass of submicroscopic particles which constituted a large part of the deposit.

Another example is afforded by a phosphate sludge from one of the newest high-pressure units in Limbo Station. The microscopic examination included in Table III listed magnesium phosphate as a minor constituent. From the X-ray diffraction pattern it could only be concluded that both calcium and magnesium phosphate were present in appreciable amounts along with magnetic iron oxide and metallic copper. Quantitative analysis established, however that magnesium phosphate was the major constituent of the deposit, thereby influencing to an important degree the recommendations for modification in treatment. The apparent failure of the microscopic examination to assay the sample correctly was due to the fact that the bulk of the sample consisted of submicroscopic particles of calcium and magnesium phosphates so thoroughly intermixed that no optical measurements could be made to estimate the relative amounts, while the larger crystals of magnesium phosphate representing only a fraction of the total amount of this constituent were easily recognized.

The war has already brought new equipment and new problems to Limbo Station, and doubtless will bring more. To diagnose the new problems may take all of the cooperative common sense of the analyst, the microscopist and the X-ray specialist, sparked by the very human desire to be the first to give the right answer to each baffling case.

Acknowledgment:

Microscopic studies included in this paper were carried out by Misses Arloa McCanne and Agnes Sharp, whose keen interest and effective work is gratefully acknowledged.

[See General Discussion, p. 1308.—ED.]

THE INTERPRETATION OF ANALYSES AND PROBLEMS ENCOUNTERED IN WATER DEPOSITS

By J. A. Holmes[1] and A. O. Walker[1]

Synopsis

Deposits from water, or so-called scales, are usually analyzed by one of several methods ordinarily used for limestone or mineral analysis. When this analysis is completed, the various radicals are combined according to some arbitrary system, based on experience, in an effort to determine just what compounds are present in the scale and thus locate the cause of the scale formation. These are called "probable combinations" and the word probable is about the only thing that can be definitely relied upon by such a system—especially if the scale happens to be a complex silicate or phosphate.

This paper is an attempt to describe the problems encountered when investigating a scale sample and to describe some of the methods used to identify the components in the scale.

In general, there are now four different methods of investigating a deposit:

1. Chemical analysis
2. Ordinary microscopic examination
3. Petrographic examination
4. X-ray diffraction examination.

Chemical Analysis:

The chemical analysis method is the one most used, and is still necessary even if other methods are used, because it gives clues for materials to be looked for by the microscopist and is an added check on results. For ordinary scales such as carbonates, sulfates, iron, or those relatively simple in structure, the chemical analysis is usually sufficient in itself. The advisabilty of combining the various components into "probable combinations" is one that should be decided by the A.S.T.M., and, if advisable, then some method should be adopted that can be universally used. Advantages of the chemical analysis method are that few laboratories have available equipment or personnel for other methods. One of the disadvantages of chemical analysis is that there are sometimes several different combinations that can be made, and this method does not show which one is correct. This method gives exact percentages of various elements present whereas other methods only approximate percentages and often miss the presence of materials present in minute quantities.

In the chemical analysis method, the most common source of error by inexperienced analysts has been in the determination of alumina. In a scale containing iron and phosphate, the determination of alumina is a procedure open to serious errors, unless certain precautions are taken. The alumina is usually determined by difference in the R_2O_3 group. The percentage of P_2O_5 and Fe_2O_3, which are determined independently before the R_2O_3 is precipitated, is subtracted from the total R_2O_3 precipitate. The difference is alumina. In order to prevent occlusion of CaO in this precipitation, it is necessary to add sufficient standard iron solution to have an excess of Fe, otherwise CaO will be precipitated as calcium phosphate and weighed as alumina. To assure complete removal of CaO, it is necessary to carry out a double precipitation of the R_2O_3 group.

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[1] Director of Service, and Chief Chemist, respectively, National Aluminate Corp., Chicago, Ill.

Another source of error is the incomplete dehydration of the silica. If the dehydration is not complete, some silica will remain in a colloidal form in the solution of iron, phosphate, and alumina, and will come down with the R_2O_3 group to be weighed as alumina.

Microscopic Examination:

The ordinary microscopic method is used mainly in looking for contamination of the deposits during sampling and handling. Pieces of metal from turbines or from too severe scraping, lint, splinters, and other materials may throw off other methods of analysis. This method is also very helpful in observing the various laminations or layers of deposits and in segregating these layers. The density or compactness of the deposit is also observed by this method. It is surprising the variety of formations one compound can have that can be seen by the ordinary microscope.

Not all of the constituents of water deposits are mineral in nature—some being composed of bacteria, fungi, or algae. Many analysts have been confused by the presence of these organisms in deposits (mostly of a soft, slimy nature). Since these organisms are composed of at least 90 per cent water, chemical analysis will show a high moisture loss, or, if the samples are allowed to dry out in shipment, these organisms may not show up at all in the chemical analysis. Even though the samples show a loss on ignition, there is no way to differentiate between organic material due to organisms or that due to natural vegetation or other sources. Microscopic examination is the only way to analyze these deposits and all iron scales or any products of corrosion should always be thus investigated.

This method is also used to test unknowns by watching color changes or crystal formations after adding reagents to the surface of a deposit.

Petrographic Examination:

The third or petrographic method makes use of the polarizing microscope which measures optical properties of the crystals. This method is limited to those compounds that form definite crystals above a certain size. It has enabled the analyst to identify many compounds in scale. It is especially valuable for rapidly obtaining the approximate composition, especially when the source and the conditions under which the scale was formed is known. This saves long and tedious chemical analysis. Disadvantages of this method are that there are still compounds of crystalline structure too small to be identified, and it does not identify amorphous materials. Considerable experience and a fairly expensive microscope are also necessary.

X-ray Diffraction Examination:

The fourth or X-ray diffraction method has a much wider range than the petrographic method and will identify compounds of any crystalline structure if the X-ray pattern is known. At the present time, it is the last resort to identify the compounds present. It does not identify an amorphous deposit. This method requires a highly trained technician and expensive equipment.

Without this method, however, it is impossible to identify positively some components of scale. For example, systems of probable combinations may suggest combining the CaO and P_2O_5 as calcium triphosphate, whereas the X-ray shows these components definitely present as calcium hydroxyapatite or $Ca_{10}(PO_4)_6(OH)_2$. Some chemists have reported calcium phosphate and free lime in scales when they should have reported the hydroxyapatite.

IDENTIFICATION OF COMPOUNDS

The calcium carbonate and calcium sulfate scales are easily identified by the petrographic method and, in some cases, by the ordinary microscope. The unusually high birefringence of calcium carbonate makes it easily identified by examination between crossed-nicol prisms. The amount of calcium carbonate can be estimated with a fair degree of accuracy. Calcium sulfate scales are usually easily identified by the appearance of the crystals which are usually relatively large and consequently the optical properties can be readily determined.

The presence of other constituents in scales is not so readily apparent. The silicates do not always form crystals large enough for microscopic study, but a few silicates have been identified by their optical properties. The presence of the mineral xonotlite ($5CaSiO_3 \cdot H_2O$) has been identified by Clark and Bunn[2] using the X-ray diffraction method. By means of its optical properties, it has been found in some scales encountered in our laboratory. Partridge has reported the mineral radiophyllite $CaSiO_3 \cdot H_2O$. A sodium-calcium silicate, pectolite, has also been identified by Clark and Bunn.[2]

Of the sodium aluminum silicate encountered in scales, the mineral analcite has been positively identified in only a few scales by optical properties. One of these was from the blades of a low-pressure turbine and the other was from the dry spot on a tube from one of our experimental boilers, so conditions must evidently be such as to allow for crystal growth. Scales are encountered which by chemical analysis contain the elements of analcite or zeolite scales in approximately the correct proportions but there were usually no visible crystals. The X-ray method is needed to show the presence of these complex compounds.

TABLE I.—ANALYSES OF A DEPOSIT CONTAINING ONLY CALCIUM AND PHOSPHATE.

Chemical Analysis, per cent		Petrographic Analysis	X-ray Analysis
Loss on ignition	2.5	Amorphous mass, unidentifiable	Hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$
SiO_2	...		
P_2O_5	42.0		
Fe_2O_3	1.0		
SO_3	...		
CaO	50.4		
MgO	3.8		
Al_2O_3	...		
CO_2	...		

Hypothetical Combinations Without Aid of Petrographic or X-ray Analysis, per cent

Loss on ignition	0.7
Fe_2O_3	1.0
$Ca_{10}(PO_4)_6(OH)_2$	90.1
$Mg_3(PO_4)_2$	7.1
$Mg(OH)_2$	0.8

CONCLUSION: Hydroxyapatite.—In this case, X-ray analysis simply confirmatory.

TABLE II.—ANALYSES OF A DEPOSIT CONTAINING MIXTURE OF MAGNESIUM AND PHOSPHATE.

Chemical Analysis, per cent		Petrographic Analysis	X-ray Analysis
Loss at red heat	7.4	Two main constituents not identified. Crystallized material not $Mg(OH)_2$. Refractive index $\alpha 1.54$ $\gamma 1.56$	Unidentified pattern. Small amount $Mg(OH)_2$. Magnesium and phosphorus high. Upon ignition pattern shows mixture MgO (10–20 per cent) and new unidentified pattern. It is not: $Mg_3(PO_4)_2 \cdot 4H_2O$ $Mg_3(PO_4)_2 \cdot 8H_2O$ $Mg_2P_2O_7$ Probably not magnesium hydroxyapatite.
SiO_2	3.4		
P_2O_5	39.0		
Fe_2O_3	1.0		
CaO	2.3		
MgO	46.5		

Hypothetical Combinations Without Aid of Petrographic or X-ray Analysis, per cent

Loss on ignition	1.6
Fe_2O_3	1.0
$Ca_{10}(PO_4)_6(OH)_2$	4.1
$Mg_3(PO_4)_2$	69.1
$MgSiO_3$	5.6
$Mg(OH)_2$	18.4

CONCLUSION: Mainly magnesium phosphorus complex not characterized.

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[2] L. M. Clark and C. W. Bunn, "The Scaling of Boilers—Part IV Identification of Phases in Calcium Silicate Scales, *Journal*, Soc. Chemical Industry (London), Vol. 59, August, 1940, pp. 155–158.

Calcium phosphate does not form crystals large enough for microscopic study, although one may become familiar with their general appearance under ordinary microscopic examination. The X-ray method has confirmed that calcium phosphate is usually present as hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$.

Table I shows a comparison of analyses of a deposit containing only calcium and phosphate. Ordinary chemical analysis is sufficient for identification.

Table II shows a mixture of magnesium and phosphate which to date has not been positively identified.

At one time much trouble was encountered in the analysis of deposits taken from sugar-beet evaporators. The scales had a high loss on ignition and a high calcium content, but there was no negative ion to tie up with the calcium. The scales were not alkaline as calcium hydroxide would have been. Examination under the polarizing microscope revealed the presence of calcium oxalate.

A scale from a turbine blade was encountered which contained 85 per cent free silver metal. An overzealous field representative had scraped a silver-plated blade a little too well. Free particles of iron, copper, and alloys are very frequently found.

Scales containing predominantly magnesia will calculate to various magnesium silicates, magnesium oxide, and magnesium hydroxide. The magnesium hydroxide can be identified under the microscope. If magnesium oxide were present in sufficiently large particles, it would be immediately apparent due to its unusually high index of refraction, but it has never been seen in any scales in our laboratory. The magnesia is probably tied up with the silica in a complex silicate of indeterminate optical properties, due either to widely variable composition, submicroscopic, or noncrystalline character.

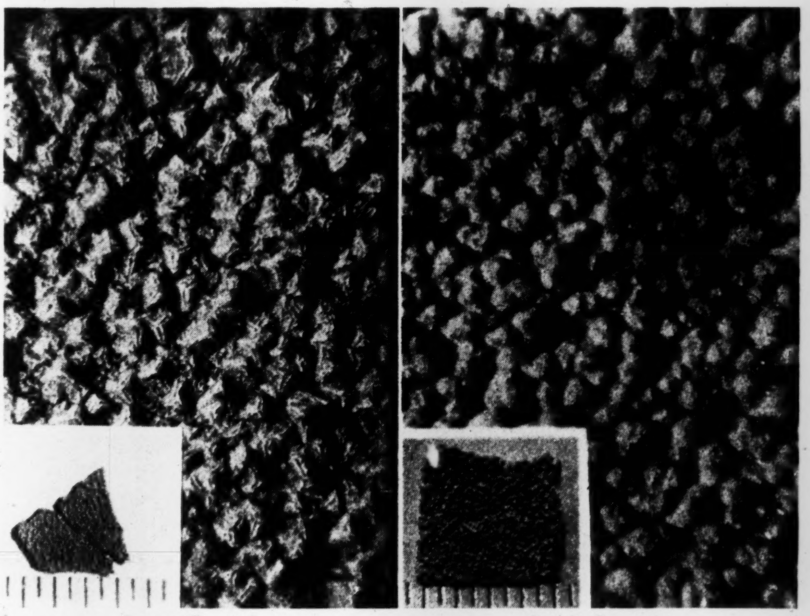
The presence of iron compounds can usually be inferred by the color of the particles. The iron often stains the crystalline substances present. The presence of crystalline iron silicates has been known, but the optical data were not determined due to the fact that the index of refraction of the crystals was above the range of the usual index liquids. However, the fact that the index was so high indicates that an iron silicate was present since the oxides of iron have not been encountered in crystalline form satisfactory for optical identification.

The presence of free metal in a scale can usually be confirmed by microscopic means. The particles, if they are large, will exhibit a metallic luster. A test with silver nitrate will also reveal the presence of copper or other heavy metals. The formation of sparkling crystals of silver immediately confirms the presence of free metal in the scale.

Considerable information can be obtained about the conditions under which scale is formed by examination with the ordinary microscope. One can distinguish between baked-on sludge and true crystal deposition. It is also useful in studying the alteration in scale structure due to different treatments, water changes, concentrations of boiler water solids, or even changes in operating conditions.

A medium power binocular microscope is the most useful, due to the fact that it furnishes stereoscopic vision and is much easier on the eyes when used for prolonged periods.

The appearance of a scale deposit can furnish a clue to the conditions under which it is laid down. Two simple examples of this are shown in Fig. 2 by a photomicrograph of a calcium sulfate scale laid down at 250 psi. and another at 25 psi. The crystals are quite different in size as well as in form. The

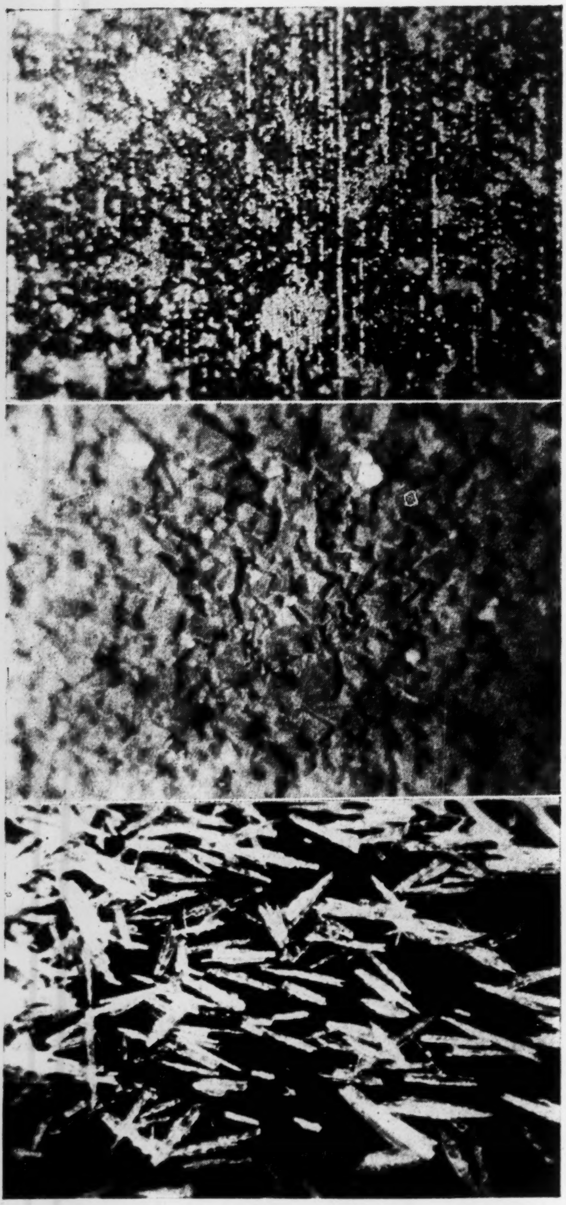


Large Crystals Small Crystals

FIG. 1.—Zeolite Type Boiler Scales of Same Type, One Large Enough To Be Partially Identified by Ordinary Microscope, Other Too Small for Optical Identification (× 50).

(*a*) 25 psi. (*b*) 250 psi.

FIG. 2.—Two Types of Sulfate Scale Formed at Different Pressures (× 10).



(*a*) Formed at 100 psi. in Area of Poor Circulation.

(*b*) Formed at 250 psi. Cool Section of Boiler.

(*c*) Formed at 250 psi. on evaporating surface.

Fig. 3.—Carbonate Crystals Formed Under Different Conditions (× 10).

scale laid down at 25-psi. pressure is composed of hexagonal crystals of calcium sulfate hemihydrate. The crystals laid down at 250-psi. pressure are anhydrous calcium sulfate or anhydrite and belong to the orthorhombic system.

In Fig. 3 are three illustrations showing a similar contrast between calcium carbonate deposits. Fig. 3 (*b*) is a scale laid down at 250 psi. while Fig. 3 (*a*) was formed in an area of poor circulation in a 100-psi. pressure boiler. Figure 3 (*b*) is composed of calcite and Fig. 3 (*a*) of aragonite. The third, Fig. 3 (*c*), indicates extremely small crystals formed directly on an evaporating surface at 250 psi.

CONCLUSION

The question usually brought up is whether there is any great value in determining the true identity of compounds in scale or is the mere determination of the various radicals or elements present sufficient. In other words, if there is calcium, magnesium, silica, iron, or alumina present, what matters other than their elimination from the feedwater? If there are corrosion products, then eliminate the corrosion by properly treating the water. In general this may be true, but this is not sufficient for the cases where the cause of the deposit is not easily determined because all treating and operating conditions appear to be perfect. Furthermore these various elements are present as definite compounds, and, such being the case, it is the job of a good analyst to identify and report their exact composition and as far as possible their physical characteristics. If this is done, then there is a much better opportunity to determine not only the source of the various components but conditions under which they are formed.

Since the average analyst must still depend on the chemical method of analysis, the Society should formulate a method of so-called probable combinations (for lack of a better term) that will fit the majority of cases and thus standardize as nearly as possible methods of reporting scale analyses. Thus, information on analyses can be exchanged more advantageously among the various chemists and engineers.

GENERAL DISCUSSION

MR. H. W. RINN.[1]—In the analysis of over fifteen hundred samples of scales and deposits from boilers and various plant equipment during the past five years, I have encountered several compounds in addition to those listed in the three papers. These are:

Atacamite—$CuCl_2 \cdot 3Cu(OH)_2$
Chalcopyrite—$CuFeS_2$
Covellite—CuS
Fayalite—Fe_2SiO_4
Goethite—$\alpha FeOOH$
—$\beta FeOOH$
Lepidocrocite—$\gamma FeOOH$
Hydromagnesite—$3\ MgCO_3 \cdot Mg(OH)_2 \cdot 3H_2O$
Hydrotalcite—$MgCO_3 \cdot 5Mg(OH)_2 \cdot 2Al(OH)_3$
Sphalerite—βZnS
Vermiculite—$(Mg, Fe)_3(Al, Si)_4O_{10}(OH)_2 \cdot 4H_2O$
Calcium iron silicate (similar to hibschite with Fe in place of Al)-$Ca_3Fe_2(SiO_4)_2(OH)_4$

The X-ray diffraction method of analysis is definitely past the "last resort" stage of development, and should be standard equipment for every general analytical laboratory. In the analysis of boiler scales I believe that in general it should be the first method used for the identification of the major constituents present. If standards are available for comparison this is quickly and easily done.

The advantages and limitations of the X-ray method are clearly presented in these papers. As the number of reliable standard patterns available for comparison increases, the usefulness of the method will also increase. In our analysis of boiler scales about a dozen patterns have been encountered for which we have no standard. Even in these cases the X-ray has given a certain amount of negative information, and by supplementary methods of analysis the elements present in the compound may be found although the exact nature of the combination is not known. Whenever one of these "unidentified" phases turns up again its presence is immediately apparent.

MR. J. A. HOLMES.[2]—I think any analyst who receives the sample of scale shown in Fig. 2 of Mr. Miller's introduction would be remiss in his report to an engineer if he did not point out the laminations in this scale. The photograph shows the bottom of the scale conforming with the circular area of the tube with this scale evidently forming in the bottom of the tube.

It is interesting to note that the laminations shown are almost parallel rather than being concentric with the tube. There are three distinct layers and these laminations, as you note, are almost straight lines across.

It is probably not up to the analyst to tell why those laminations are there—he may not be able to. But at least he should report laminations in that form to whoever is receiving a copy of the analysis, and, more than likely, send him a photograph of the scale. Of course, it is true that whoever takes the sample would probably notice these laminations, but the analyst who is looking at the scale either under a microscope or by some other close observational method would most likely see them and should show the presence of these laminations in his report.

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[1] X-ray Analyst, Spectroscopy Laboratory, The Dow Chemical Co., Midland, Mich.

[2] Assistant Vice-President, National Aluminate Corp., Chicago, Ill.

MR. C. E. IMHOFF.[3]—Table II of Messrs. Holmes and Walker's paper showing the analyses of a magnesium phosphate deposit is illustrative of the case in which identification cannot be made, due to lack of an identical standard. The diffraction analysis, however, shows that the compound is not one of the common magnesium orthophosphates ordinarily represented in the writing of chemical equations. Numerous instances of this sort of thing have been encountered in our X-ray laboratory. It is a constant reminder that many of the solid phases which develop in feedwater and steam cycles are not always those which are expected according to simple ionic reactions. It also is a constant reminder that considerable research is still necessary to describe completely the conditions of formation of these uncommon compounds.

MR. EVERETT P. PARTRIDGE.[4]—I want to bring out one point in connection with the suggestion once made that Committee D-19 should standardize methods of making probable combinations of the oxides shown by chemical analysis. In the case of a simple substance, relatively uncontaminated, interpretation might be made without specific identification; general application of any scheme for hypothetical combinations unsupported by independent evidence concerning the actual constituents would, however, almost certainly lead to error in the case of more complex deposits.

It seems to me that a substance should be reported definitely as a constituent of a deposit only when its presence is proved by either microscopic or X-ray identification together with chemical evidence.

MR. R. B. SOSMAN[5] (*by letter*).—I should like to ask whether the cristobalite (SiO_2) mentioned in Table I and in No. 7 of Fig. 4 of the paper by Partridge, Scott, and Morrison could have arrived in suspension from some original source such as silica brick; or could only have formed where found, by separation from clear water. Cristobalite is usually considered a high-temperature mineral (200 C. and higher) and is completely stable only above 1470 C., but J. W. Gruner has found it in considerable percentage in bentonite under circumstances where it seems that it must have formed by processes of every-day weathering. Yet it has never been identified in the products of weathering of any igneous rock excepting the volcanic glass from which most bentonites are derived.

MR. R. K. SCOTT[6] (*author's closure, by letter*).—The cristobalite mentioned in Table I and as No. 7 of Fig. 4 in our paper, which prompted Mr. Sosman's question, was found as a scale in the bottom of a steam drum of a boiler under the feedwater pipe. We believe that the material was originally precipitated as a silica gel. The feedwater contained 10 to 12 ppm. of silica and was fed into the boiler after reduction of alkalinity by addition of sulfuric acid; this operation could conceivably tend to precipitate dissolved or colloidal silica. The transformation to cristobalite might then have taken place by dehydration of the gel on the drum surface in the presence of the boiler-water salts as mineralizers. This boiler operated at a pressure of 150 lb., so that the temperature of the boiler water was approximately 185 C. . This is slightly lower than the 200 C. mentioned by Mr. Sosman, although the temperature of dehydration may actually have been somewhat higher than that of the boiler water, depending on the rate of heat transfer through the wall of the drum and the insulating effect of the deposit itself.

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[3] Chemical Engineer, Feedwater Treating Dept., Allis-Chalmers Manufacturing Co., Milwaukee, Wis.

[4] Director of Research, Hall Laboratories, Inc., Pittsburgh, Pa.

[5] Physics Supervisor, Research Laboratory, United States Steel Corp. of Delaware, Kearny, N. J.

[6] X-ray Specialist, Hall Laboratories, Inc., Pittsburgh, Pa.

MR. GEORGE W. MOREY[7] (*by letter*).—Cristobalite is frequently formed at high pressures of water under conditions in which it is unstable.[8] In our work we have obtained it by heating amorphous silica with water in closed vessels at from about 300 to 400 C. On longer treatment or at higher temperatures or in the presence of alkali, quartz will form, and under these conditions cristobalite will be changed to quartz.

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[7] Geophysical Laboratory, Carnegie Institution of Washington, Washington, D. C.

[8] George W. Morey and Earl Ingerson, "The Hydrothermal and Pneumatolytic Alteration and Synthesis of Silicates," *Economic Geology*, No. 32, p. 607 (1937).

SUMMARY OF PROCEEDINGS OF THE BUFFALO SPRING MEETING

SYMPOSIUM ON PAINT

SYMPOSIUM ON POWDER METALLURGY

The 1943 Spring Meeting of the Society was held in Buffalo, N. Y. at the Hotel Statler on Wednesday, March 3, 1943 in conjunction with the spring group meetings of A.S.T.M. committees. The Spring Meeting consisted of two formal technical symposiums, one on paint and the other on powder metallurgy. Both symposiums were sponsored by the Buffalo Committee on Arrangements under the direction of B. L. McCarthy of the Wickwire Spencer Steel Co., Chairman; W. H. Lutz, of Pratt & Lambert, Inc., Vice-Chairman; and T. L. Mayer of Buffalo Public Library, Secretary.

Symposium on Paint:

Two sessions were devoted to the Symposium on Paint—morning and afternoon sessions. The symposium was developed by W. H. Lutz, Vice-Chairman of the Buffalo Committee on Arrangments, with the assistance of C. H. Rose of the National Lead Co., on behalf of the Society's Committee E-6 on Papers and Publications. The symposium consisted of the following eight papers:

"Summary of Protective Coatings Raw Materials Situation," by A. C. Goetz, Consultant, Paint, Varnishes, and Lacquer Unit, Protective Coatings and Materials Section, War Production Board.

"Drying Oil and Fatty Acid Development," by A. Schwarcman, Research Chemist, Spencer Kellogg and Sons, Inc.

"Surface Coating of Opaque Pigments," by Roscoe H. Sawyer, Pigments Dept., E. I. du Pont de Nemours & Co., Inc.

"Surface Coating of Calcium Carbonate Extenders," by E. W. Boughton, Manager, Paint Dept., R. T. Vanderbilt Co., Inc.

"Emulsion Paints," by Carl Iddings, Vice-President and General Manager, Prescott Paint Co., Inc.

"Protective Concealment Paints," by Paul O. Blackmore, Director of Products Engineering and Development, Products Engineering and Development Laboratory, Ault & Wiborg Corp.

"Blackout Paints," by John J. Bradley, Jr., Chief Chemist, Bird and Sons, Inc. Introduction to paper, by J. C. Moore, Superintendent, Paint Plant, Sinclair Refining Co.

"Luminous Paints," by G. F. A. Stutz, Technical Department, The New Jersey Zinc Co. (of Pa.).

The presiding officer of the sessions was K. J. Howe, Vice-President, Thibaut & Walker Co.

Symposium on Powder Metallurgy:

The Symposium on Powder Metallurgy, held as an afternoon session, was developed by B. L. McCarthy, Chairman of the Buffalo Committee, with the assistance of H. S. Rawdon, Chief, Division of Metallurgy, National Bureau of Standards, on behalf of the Society's Committee E-6 on Papers and Publications. The symposium consisted of the following seven papers:

"The Fundamentals Necessary to Apply Power Metallurgy," by Charles Hardy, President, Hardy Metallurgical Co.

"The Effect of Pressure on the Properties of Compacts," by Clarence W. Balke, Fansteel Metallurgical Corp.

"The Influence of Homogenization Upon the Physical Properties of Copper-Nickel Powder Alloys," Frederick N. Rhines, Assistant Professor of Metallurgy, Member of Staff of the Metals Research Laboratory, Carnegie Institute of Technology; and Russell A. Meussner, Second Lieutenant, United States Army.

"The Effect of Particle Size on the Shrinkage of Metal Compacts," by P. R. Kalischer, Chemical and Metallurgical Dept., Research Laboratories, Westinghouse Electric and Manufacturing Co.

"Alloy Powders," by T. H. Lashar, Chief Metallurgist, Wood Ridge Division, F. W. Berk Co., Inc.

"Metal Powder Friction Materials," by J. F. Kuzmick, Assistant Director of Research, Metal Powder Laboratory, Stevens Institute of Technology.

"The Hot Pressing of Metal Powders," by W. N. Pratt, Research Engineering Dept., Jones & Lamson Machine Co.

The presiding officer of the symposium was A. J. Langhammer, President, Amplex Division, Chrysler Corp.

The evening session of the spring meeting consisted of the spring meeting dinner held under the sponsorship of the newly organized New York - Ontario A.S.T.M. District Committee. The principal speaker at the dinner was Captain A. J. Wellings, Director, Material Inspection Administration, U. S. Navy Department, Washington, D. C.

The papers in both of the above symposiums elicited discussion which contributed greatly to the information contained in the papers. The papers of the two symposiums complete with discussion have been issued by the A.S.T.M. as separate publications entitled, respectively, "Symposium on Paint," and "Symposium on Powder Metallurgy." The nature of the subject covered in the paper by A. C. Goetz, presented at the Symposium on Paint, namely, statistics on available materials and substitution problems, dictated publication as promptly as possible. The paper was accordingly published in A.S.T.M. BULLETIN, No. 121, March, 1943, p. 15. It is not, therefore, included in the Symposium issued as a separate publication.

The three technical sessions were very well attended, there being over 300 persons at each session.

SUBJECT INDEX

VOLUME 43

A

Abrasion.

Strength of Concrete as Related to Abrasion of the Blast-Furnace Slag Used as Coarse Aggregate—Fred Hubbard and H. T. Williams, 1088. Discussion, 1095.

Accelerated Testing.

See also **Aging Tests; Corrosion; Weathering**

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